Direct Measurement of Adsorbant Capacity and Function Using In-Situ Raman Spectroscopy

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Adsorbents are widely used in many industries, ranging from gas processing and storage through to water purification. Increasing the performance of adsorbent-based processes is often achieved through improved adsorbent materials. Therefore, measurement techniques to characterise adsorption are key to the development of novel materials. However, conventional volumetric and gravimetric techniques suffer from a number of technical limitations that restrict their range of application, including operating pressure and temperature, measurement time, and minimum sample size. Moreover, these macroscopic techniques indirectly measure sorption through the average response of a sample, making them insensitive to material heterogeneity and structure and thus requiring complementary techniques to study microscopic behaviour such as material-phase transformations. We present a new approach based on in-situ Raman microscopy, including the experimental methodology and analsyis framework, which can be used to directly and quantitatively characterise adsorption capacity and qualitatively investigate adsorption mechanisms in materials. To demonstrate this technique, we present adsorption capacity measurements of CO2 in a 10 micro-gram sample of a commercial silica gel. The results, measured at 30 °C and at pressures up to 3 MPa, are validated using conventional volumetric and gravimetric techniques. Additionally, the pressure-response function of a gate-opening zeolitic imidazolate framework (ZIF-7) has been studied, with qualitative agreement between the pressure-induced response of the material's structure observed with Raman spectroscopy and the gravimetrically-measured adsorption capacity. This combination of direct sorption and material characterisation for small samples presents a unique capability for the rapid development and screening of adsorbent materials.