HIGH PRESSURE ADSORPTION: SPECIALIZED EQUIPMENT AND MEASURMENTS

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Since decades adsorption is one of the frequently used processes for gas separation and cleaning. Mainly applied in pressure swing adsorption plants from two up to nine beds they are normally used in a moderate pressure range below 10 bar. But especially under high pressure atmospheres adsorption is very interesting due to higher relative loading capacities, better steric effects and higher selectivity.

For determining the adsorption properties of pure gases under high pressures several measuring instruments exist. For pressures up to 50 bar volumetric/manometric instruments are widely used and sold by several manufacturers, for higher pressures up to 400 bar the most accurate tool is a magnetic suspension balance. But in industrial as well as lab applications only in very few cases (i.e. H_2 storage) pure gases are used, so that selective mixture adsorption measurements need to be performed. Therefore a prototype of a such a measuring instrument will be presented and some measured selective mixture adsorption isotherms will be compared to IAST-calculated isotherms based on pure gas isotherms.

In addition to traditional sorbent materials, such as activated carbons or zeolithic molecular sieves, many new, highly specialised sorbent materials are currently under development, including metal organic frameworks (MOFs). Due to the complicated synthesis with low yields, only small quantities of MOFs are manufactured, and very few materials are commercially available. This again adds to the challenge for the necessary, accurate sorptive characterisation: precise measurements at very small sample quantities, down to few milligrams or even less, under high pressures and elevated temperatures. Therefore a prototype of a new magnetic suspension balance with substantially higher resolution than the current state of the art will be presented. It is the first such device which measures mass increase of samples without being coupled to a micro balance with a currently achieved mass resolution of $1x10^{-7}$ g, with potential to increase this further to $2x10^{-8}$ g, all under conditions up to 250 bar and temperatures up to 200°C. Measurements and comparison to other data at high pressures of several MOFs of Basolite and Cr-MIL101 families, which have potential for use in air separation and biogas purification, will be presented.

Other novel materials currently under investigation are SILPs, an (ionic) liquid phase brought on a solid porous carrier material. For determination of such materials first of all the adsorption properties of the liquid itself needs to be investigated. With standard instruments this takes a very long measuring time due to the diffusion controlled absorption process. Therefore very special gravimetric instrument, which is currently still under development, will be presented that allows forced flow through liquid phases under high pressure conditions with an optical determination of the exchange surface area. Thus it is possible to determine the convective mass transfer coefficent.

ISBN 978-963-313-179-4