

Combined calorimetric and manometric measurements for the study of sorption properties of porous materials

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The gas sorption Sievert's technique has proven to have many advantages for the evaluation of the ad- or ab- sorbed amount of gas by porous materials in a wide range of temperature and pressure. In addition, there is a total freedom in the size and shape of the sample holder in the volumetric technique, enabling the coupling of techniques and in-situ measurements of various chemical and physical parameters. X-rays and neutrons diffractometers, gas chromatographs or mass spectrometers have already been successfully tested and allow having simultaneous PCT isotherms and kinetic measurement with structural or gas composition data.

The thermodynamics of the adsorption are essential for the practical application and among all the heat of adsorption (or desorption) is a key parameter. Practically there are two ways to determine it. The first one is an indirect method, where it is derived from adsorption isotherms at different temperatures. The second one is a direct method, where the enthalpy is measured via calorimetric techniques. When used on its own, the biggest disadvantage of calorimetry is that it gives a heat output per mole of solid sample and not per mole of gas. The combination of manometric technique (to quantify the amount of hydrogen absorbed/released) and calorimetry was successfully applied to overcome this issue and the direct measurement of enthalpy of formation per mole of gas was reported [1-3].

The presentation will give some new results on combinations of calorimetric and volumetric technique, especially on MOF-5, selected as an example of Metal Organic Framework that is available commercially. But also on different other porous materials such as amine modified mesoporous silica and hydrotalcite based catalysts. It will give an overview of the state-of-the art possibility of combined calorimetric analysis together with the Sievert's technique.

[1] M. R. Mello, D. Phanon, G. Q. Silveira, P. L. Llewellyn, C. M. Ronconi, *Microporous and Mesoporous Materials* 143 (2011) 174–179

[2] A. Auroux et al, "Calorimetry and Thermal Methods in Catalysis", Springer Series in Materials Science (2013), Vol. 154

[3] R. Bulanek, K. Frolich, E. Frydova, P. Cicmanec, *Top Catal* 53 (2010) 1349–1360