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Supercritical fluid activation and in-situ adsorption-microcalorimetric system to directly measure the gas adsorption amount, heat and kinetic data

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Porous materials play a vital role in industrial and environmental systems due to their diverse applications. The adsorption capacity and associated heat during this process are crucial parameters for evaluating the performance of these materials. Achieving high sensitivity and accuracy is of utmost importance in adsorption and microcalorimetry instruments. As a result, researchers continuously strive to enhance the precision and repeatability of measurement results by employing various theoretical approaches to calculate the heat of adsorption.

In this work, we have designed a system for activating porous materials directly using supercritical extraction. The system involves mixing supercritical carbon dioxide with organic solvents and directly introducing them into the sample chamber to achieve rapid and efficient activation of porous materials with ultra-high surface area¹. Additionally, we present a novel system comprising a Tian-Calvet microcalorimetry apparatus equipped with an automatic gas adsorption measuring unit or chemisorption instruments for in-situ measurement of physisorption and chemisorption heat, respectively². To validate its feasibility and accuracy, we conducted experiments using different gases such as CH₄, CO₂, and C₃H₈. The obtained results effectively demonstrate the instrument's accuracy and convenience for measurements. Furthermore, this instrument allows us to accurately measure the evolved energy even at extremely low adsorption capacities, commonly referred to as "the zero-point adsorption heat." This valuable information sheds light on the most active sites within porous materials, thereby contributing to a better understanding of their properties.

References:

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