Combination of adsorption volumetry and calorimetry in a novel sensor gas calorimeter

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Introduction

The design of technical adsorption processes is mainly based on measurements of pure gas isotherms and breakthrough curves. Since temperature strongly influences the breakthrough curves modeling and design of adsorption processes requires a precise knowledge of the heat emitted during adsorption. This heat of adsorption is a function of coverage, so a simultaneous measurement of adsorption enthalpy and load is desirable. This project aims at developing a measurement technique for the coupling of calorimetric and volumetric equilibrium measurements in one device.

Experimental Methods

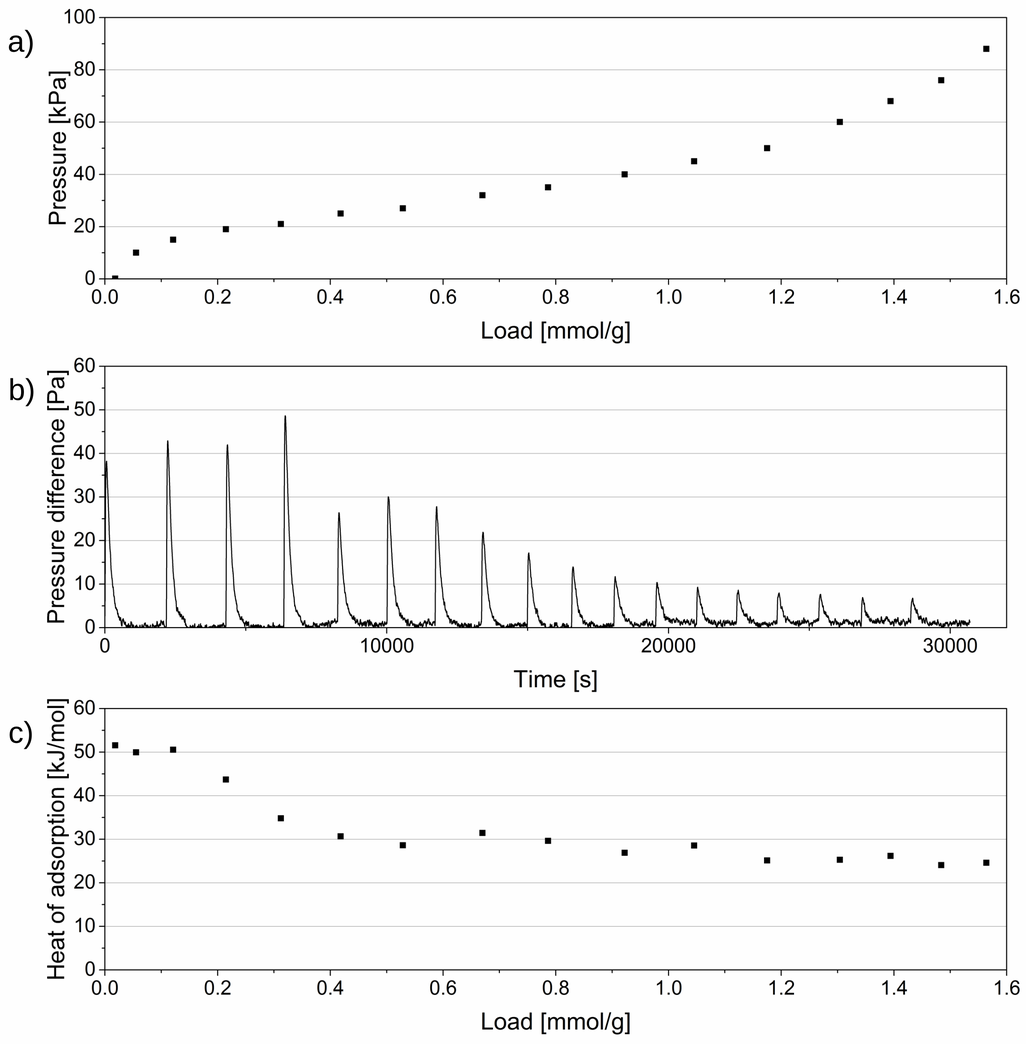

Fig.1: Analysis of measurement results a) isotherm; b) time-dependent pressure difference curve; c) load-dependent heat of adsorption

<u>isotherm:</u>

- volumetric adsorption measurement (BELSORP-max)
- cumulative measuring principle

<u>time-dependent pressure difference curve:</u>

- calorimetric measurement
- measuring pressure difference between both sensor gas volumes
- each pressure difference peak represents an adsorption step

<u>load-dependent heat of adsorption:</u>

- coupling of volumetric and calorimetric measurement
- determination of heat of adsorption for each adsorption step
- time and cost saving measuring principle due to simultaneous measurement

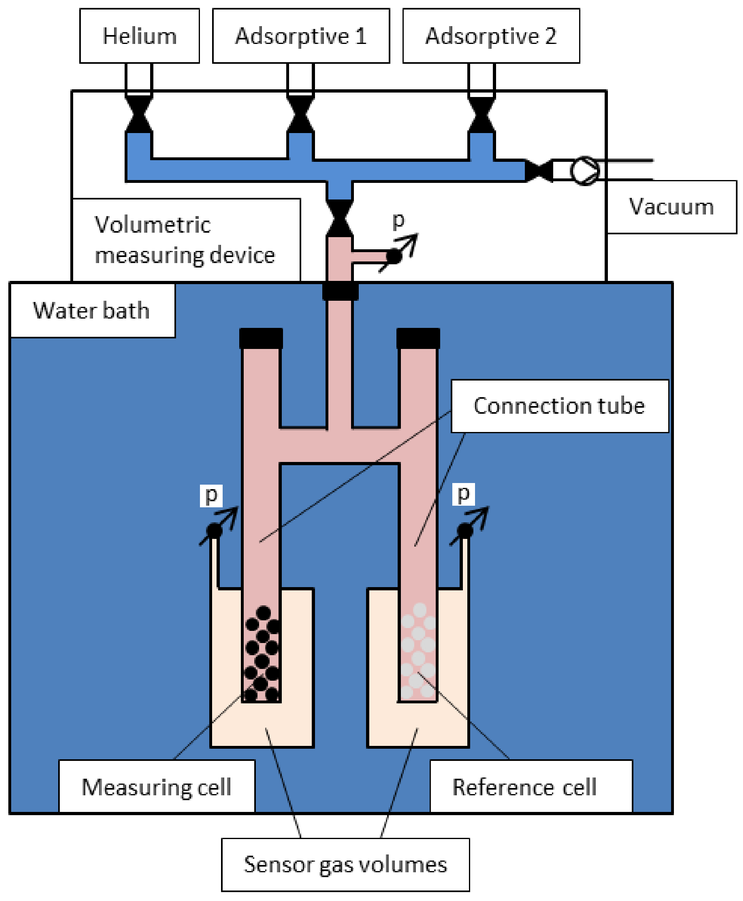

Fig.2: Experimental setup

Results and Discussion

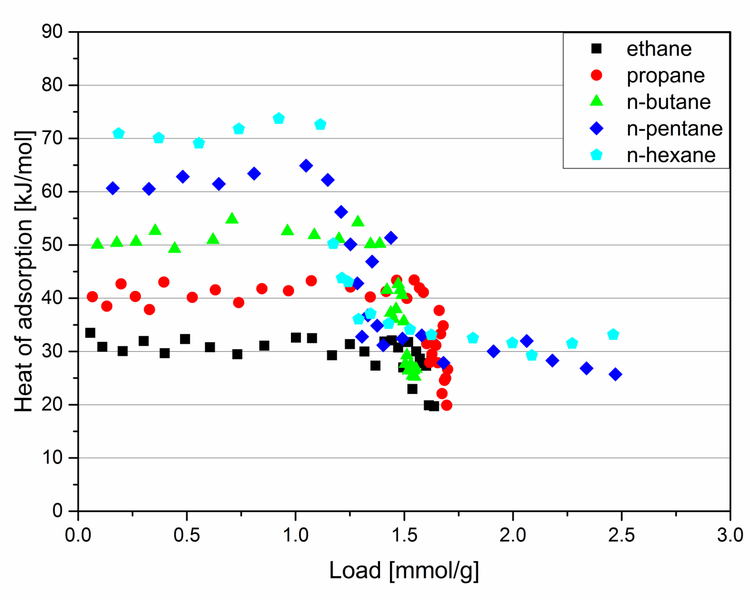

Fig.3: Load-dependent heat of adsorption of n-alkanes on ZSM-5 zeolite HiSiv 3000

- heat of adsorption (Δh_{Ads}) generally increases with chain length and polarizability
- constant Δh_{Ads} indicates a homogeneous surface
- significant decrease of Δh_{Ads} at high load by capillary condensation in the binder material

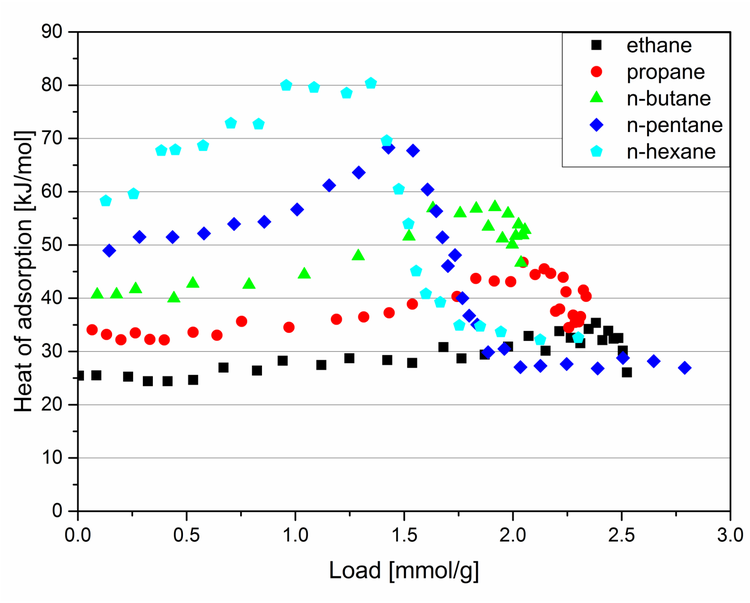

Fig.4: Load-dependent heat of adsorption of n-alkanes on faujasite zeolite 13X-APG

- Δh_{Ads} increases with load due to lateral adsorptive-adsorptive interactions
- larger load-dependent increase of Δh_{Ads} with increasing evaporation enthalpy of adsorptive

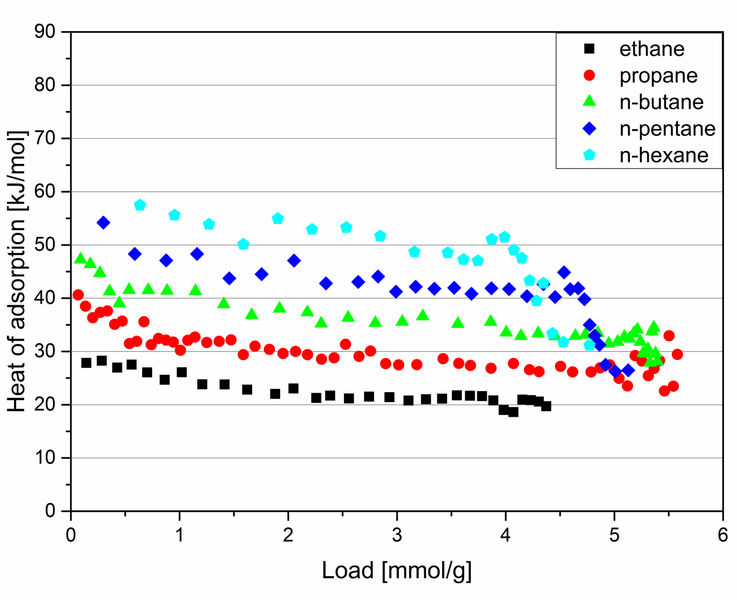

Fig.5: Load-dependent heat of adsorption of n-alkanes on activated carbon Norit R1 Extra

- decreasing Δh_{Ads} due to heterogeneous surface
- significant decrease of Δh_{Ads} at high load due to capillary condensation in mesopores

Resume and Prospect

In order to investigate the load-dependent heat of adsorption, a sensor gas calorimeter was successfully developed which allows a simultaneous measurement of calorimetric and volumetric data. As an example, the load-dependent heat of adsorption of the homologous series of n-alkanes from C2 to C6 on zeolites and activated carbon is shown.

Heats of adsorption between 20 and 80 kJ/mol were found. Based on the load-dependent heat of adsorption, an energetic characterization of the sorption systems will be carried out. Combining capacity and caloric data allows a more sophisticated analysis of surface chemistry and pore structure.

Acknowledgments

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