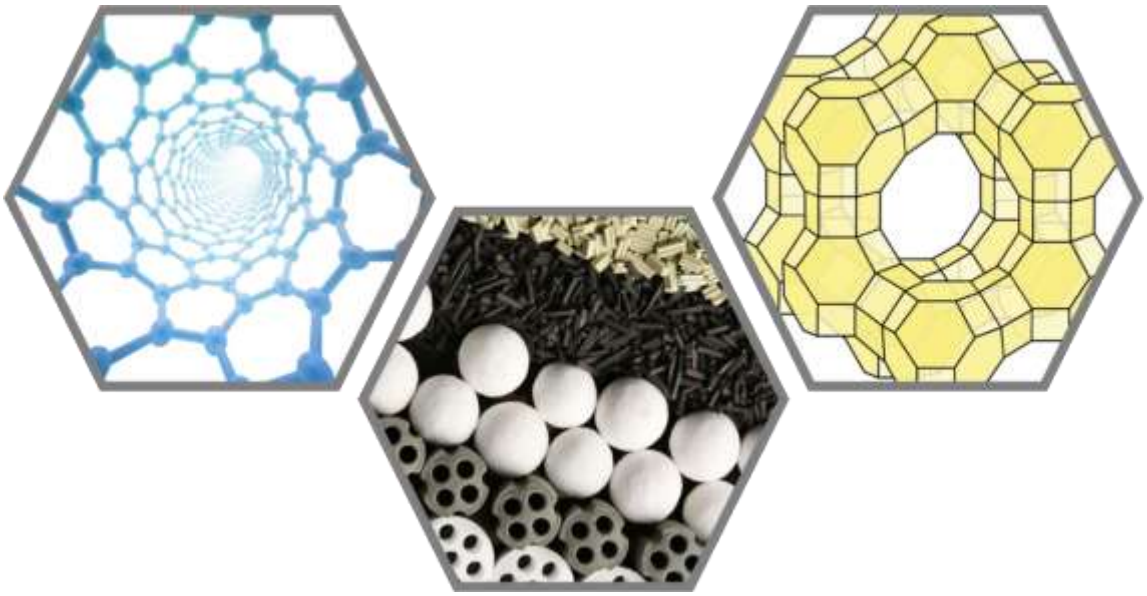
3P Adsorption Week
Adsorption & Characterization of Porous Solids



Conference on gas adsorption for surface and pore characterization

Date: 14/05 & 15/05/2024
Time Zone: UTC +2
Venue: Vienna House Easy Leipzig, Germany or online (MS Teams)
Organizers: Institute for Non-Classical Chemistry (INC) & 3P Instruments

14th May 2024

Time	Name	Affiliation	Title
8.30	**Registration**		
8.55	Dietmar Klank	3P Instruments, DE	Welcome and opening remarks
9.00	Sebastian Ehrling	3P Instruments	Technical developments in manometric sorption methods for surface and pore analysis
9.45	Marcus Lange	INC	Development of a comprehensive measurement system for membrane separation properties
10.15	**Coffee break**		
10.45	Andreas Wagner	Helmholtz Dresden Rossendorf	Positronium-annihilation spectroscopy probes open and close porosities
11.15	Jan Demel	Czech Academy of Sciences	"Activated Borane" – A Porous Borane Cluster Network
11.45	Andreas Hahn	ZetA Partikelanalytik GmbH	Surface area and pore size as key parameters on removal performance of vertical flow constructed wetlands
12.15	**Lunch break**		
13.45	Desirée Leistenschneider	Universität Jena	N-rich carbon materials and their interaction with water
14.15	Francesco Walenszus	3P Instruments	Studies of sorption equilibria with unique stability of temperature and relative pressure
14.45	**Coffee break**		
15.15	Prof. Arvind Rajendran (online)	University of Alberta	Dynamic column breakthrough methods to understand competitive equilibria for adsorptive CO_2 capture
16.00	Tai Nguyen	Svante	Characterization of laminate CALF-20 and its process intensification for capturing CO_2 from point sources
16.30	**Poster & Beer**		

15th May 2024

Time	Name	Affiliation	Title
9.00	Jens Möllmer	INC	From laboratory to industrial scale and back - examples in the field of adsorptive separation processes
9.45	Prof. Pantelis N. Trikalitis	University of Crete	Intriguing gas/vapor sorption properties in highly flexible rare-earth MOFs
10.30	**Coffee break**		
11.00	Fabian Schönfeld	3P Instruments	New aspects regarding measurement accuracy, flexibility, ease of maintenance and sustainability of sorption analyzers
11.30	Aleksandra Marcinek	CarboTech AC GmbH	How CMS structure affects the PSA performance
12.00	**Lunch break**		
13.30	Prof. Camille Petit (online)	Imperial College London	Technological considerations for the advancement of adsorption-based direct air capture
14.00	Tobias Horn	Airbus	Countering climate change – Filtering out CO_2 from the atmosphere and making it versatile
14.45	**Coffee break**		
15.15	Sven Helle	Brandenburgische Technische Universität Cottbus-Senftenberg	A Comparative Investigation of Surface Interactions of Porous Materials using Nitrogen and Argon Adsorption
15.45	Christian Teicht	Fraunhofer-Institute for Chemical Technology	An easy-to-use modification of the potential theory of adsorption
16.15	**Closing remarks**		

Technical developments in manometric sorption methods for surface and pore analysis

Sebastian Ehrling, Robert Eschrich, Dietmar Klank 3P Instruments

In the last decades advancements in manometric sorption methods have significantly elevated the precision and efficiency of surface and pore analysis in materials science. Especially the integration of cutting-edge technologies, such as high-resolution pressure transducers and automated data acquisition systems, facilitating real-time monitoring of sorption processes. Novel approaches in sample preparation and instrument calibration have been implemented to enhance the accuracy of surface area and pore size determinations. Furthermore, the incorporation of advanced computational models has enabled a deeper understanding of complex sorption behaviors. These technical developments collectively contribute to the refinement and reliability of manometric sorption methods, establishing them as indispensable tools for comprehensive surface and pore analysis in diverse materials.

Development of a comprehensive measurement system for membrane separation properties

Marcus Lange, INC Leipzig

The lecture will introduce a new membrane cell (MOMT - multi-operable membrane test cell) that can be used to investigate the sorption and permeability properties of flat membranes, textiles or filters of any kind. The full functionality of the MOMT arrangement and its integrability into existing measuring systems or test set-ups will be presented using several examples based on variation of experimental conditions and adsorptives/adsorbents studied.

Positronium-annihilation spectroscopy probes open and close porosities

Andreas Wagner, Helmholtz Dresden Rossendorf

Tba.

"Activated Borane" – A Porous Borane Cluster Network

Jan Demel, Czech Academy of Science

In 2022 our laboratory has introduced activated borane as a new class of microporous polymers. It is formed by co-thermolysis of open borane clusters and small organic molecules such as toluene or cyclohexane. The amorphous structure of activated borane is probably formed by closed (closo) borane clusters connected by organic linkers forming a porous framework.
In the present talk, I will discuss their properties and structural variability.

Surface area and pore size as key parameters on removal performance of vertical flow constructed wetlands

Andreas Hahn, ZetA Partikelanalytik GmbH

Constructed wetlands for waste-water treatment have been increasingly recognised worldwide in recent years and have more than five decade history in Europe. The removal principle is commonly described as a filter effect and therefore fluviatile sands are employed as they are efficient filter materials, chemically relatively inert, connected to low costs and easily available - but with relatively short life time due to clogging from growth of biofilms.
In case of porous lava sands are employed as filter media instead of non-porous fluviatile sand, high removal rates of nitrate, nitrite as well as organic contaminants are observed - year after year without clogging.
Investigation of surface area and pore system by means of gas sorption and mercury intrusion shows that lava sands exhibit a broad spectrum of accessible pores - helping to understand the mechanism of waste-water treatment.
While zeolite-derived micropores quickly filled during operation, larger mesopores are still accessible after years of operation. These pores enable small molecules to access a relatively high surface area - especially in comparison with fluviatile sands - and thus help to stabilise biofilms. In conclusion in this case the process of waste water treatment can be seen as a biocatalytic-fixed bed reactor system.

N-rich carbon materials and their interaction with water

Desirée Leistenschneider, Universität Jena

Carbons have become an integral part in the field of energy storage materials. Through functionalization with heteroatoms such as N, O or B, their interaction with Lewis bases, such as water, can be tailored towards their final application. Therefore, water adsorption experiments are carried out to understand the role of different functional groups within N-rich carbon materials. Changes within the materials due to water adsorption are characterized using XPS and IR spectroscopy.

Studies of sorption equilibria with unique stability of temperature and relative pressure

Francesco Walenszus, Dietmar Klank, Sebastian Ehrling 3P Instruments

Key highlights of our work include the development of a unique experimental apparatus capable of maintaining constant and reproducible temperature and therefore pressure conditions. This novel setup allows us to investigate sorption equilibria with unprecedented precision, offering a deeper understanding.

Dynamic column breakthrough methods to understand competitive equilibria for adsorptive CO_2 capture.

Nicholas Wilkins, Noelie Constant, Gwyneth Liske, James Sawada, Arvind Rajendran University of Alberta

The last few years have seen a dramatic increase in research focussed on CO_2 capture. We have seen several new adsorption processes scaled up for post-combustion, pre-combustion CO_2 capture and direct air capture (DAC). Particularly, the success of Calgary framework-20 (CALF-20), the first metal-organic framework (MOF) to be scaled up for industrial CO_2 capturer and with the possibility that it could be manufactured in the hundreds of tonnes scale, has reinvigorated the interest in novel materials. From the process side, new ways of operating, especially processes that use direct-stem injection for regeneration, have created new opportunities for engineering research. The success of these processes is underpinned by the nature of binary interactions of gas/gas, gas/water and gas/steam on rather exotic materials that have not been studied in the literature. However, a literature scan shows very little data and, hence, very little understanding of the binary adsorption of the pairs mentioned above. Hence, in the past years, we have been focused on developing experimental techniques to measure binary adsorption.

In this presentation, we will highlight recent developments in adsorption-based CO_2 capture and dynamic column breakthrough (DCB) to quantitatively measure competitive equilibria on small samples (100s of milligrams). We will highlight the extensive characterization of CO_2/N_2, CO_2/H_2O and CO_2/steam competition on CALF-20 and other CO_2 capture adsorbents measured in our lab. The breakthrough curves open a fascinating window into wave propagation dynamics and reveal features that have possibly not been seen in classical adsorbents, such as zeolites and carbons. We will also take the opportunity to provide a short tutorial-type introduction to DCB methods to highlight how they can (and should not) be used to estimate adsorption equilibrium.

Characterization of laminate CALF-20 and its process intensification for capturing CO_2 from point sources

Tai Nguyen, SVANTE

CALF-20 is a Metal-Organic Framework (MOF) adsorbent developed and commercialized in manufacturing by Svante. The single component equilibrium and the dynamic column breakthrough study of CO_2, N_2 and H_2O on laminate CALF-20 were examined at various compositions and conditions. The data have been used to develop and optimize the Rapid Cycle Thermal Swing Adsorption (RCTSA) cycle for Svante's CO_2 capture technology. Large-scale commercial manufacturing of the CALF-20 adsorbent has been achieved, and the material has been tested in an 1TPD CO_2 capture pilot plant and demonstrated satisfactory performances [1]. This process intensification directly supports CO_2 capture processes and mitigates risks associated with scaling up CO_2 capture technology from adsorption-based processes.

[1] Ghaffari Nik, O., Liu, A., Moroy, B., Barbot, C., Henkel, B., Llewellyn, P., ... & Hovington, P. (2023). Rapid Cycle Temperature Swing Adsorption Using Calf-20 MOF Structured Adsorbent for Cement Carbon Capture. Andrew and Moroy, Berenice and Barbot, Claire and Henkel, Brett and Llewellyn, Philip and Voysey, Stephanie and Hovington, Pierre, Rapid Cycle Temperature Swing Adsorption Using Calf-20 MOF Structured Adsorbent for Cement Carbon Capture (February 6, 2023).

From laboratory to industrial scale and back - examples in the field of adsorptive separation processes

Jens Möllmer, INC Leipzig

The presentation shows which questions arise in the operation of technical adsorbing units and how these can be answered based on extensive laboratory tests. Practical examples such as the removal of VOCs or drying of natural gas will be used to illustrate procedures and solutions..

Intriguing gas/vapor sorption properties in highly flexible rare-earth MOFs

Pantelis N. Trikalitis, University of Crete

Flexible MOFs represent an important class of functional porous materials for numerous applications including gas/vapor storage, separation and sensing, among other. We will present our recent development[1-3] in flexible rare-earth MOFs where structural dynamics and gas/vapor sorption properties can be systematically tuned, affording materials with a unique gas-trapping behavior and high selectivity towards VOCs.

References

(1) Angeli, G. K.; et. al, Continuous Breathing Rare-Earth MOFs Based on Hexanuclear Clusters with Gas Trapping Properties. *J. Am. Chem. Soc.* **2021,** *143* (27), 10250-10260.

(2) Loukopoulos, E.; et. al, Accessing 14-Connected Nets: Continuous Breathing, Hydrophobic Rare-Earth Metal Organic Frameworks Based on 14-c Hexanuclear Clusters with High Affinity for Non-Polar Vapors. *ACS Applied Materials & Interfaces* **2022,** *14* (19), 22242-22251.

(3) Loukopoulos, E.; et al, Reticular Synthesis of Flexible Rare-Earth Metal-Organic Frameworks: Control of Structural Dynamics and Sorption Properties Through Ligand Functionalization. *Chem. Eur. J.* **2023,** *n/a* (n/a), e202302709.

New aspects regarding measurement accuracy, flexibility, ease of maintenance and sustainability of sorption analyzers

Fabian Schönfeld, Dietmar Klank, Sebastian Ehrling 3P Instruments

This presentation encapsulates a comprehensive overview of recent developments in sorption analyzers, highlighting their refined measurement accuracy, expanded flexibility, simplified maintenance protocols, and commitment to sustainability. Researchers, engineers, and industry professionals stand to benefit from insights into the evolving landscape of sorption analysis, fostering advancements in scientific understanding and practical applications.

How CMS structure affects the PSA performance

Aleksandra Marcinek, CarboTech AC GmbH, Essen/Deutschland

The structural characterization of Carbon Molecular Sieves remains a challenge due to a complex network of partially restricted pores exhibiting substantial diffusion limitations. Yet, a unique arrangement of the deposited pyrocarbon layer within the porous carbon particle dictates the adsorption selectivity of CMS and therefore the overall process performance of kinetically-controlled separations such as N_2/O_2 or CH_4/CO_2. For this reason, the process-based evaluation of CMS quality is still considered the most foolproof method with respect to a particular application, as it directly provides data about the separation effectiveness in authentic operating conditions. However, combining process data with an insight into the material structure allows the design of novel adsorbents in favour of PSA systems exhibiting reduced CAPEX and/or OPEX. The characterization results of commercial CMS together with N_2-PSA performance are presented and discussed. Moreover, the guidelines for material selection for specific process conditions or product purity requirements are given.

Technological considerations for the advancement of adsorption-based direct air capture

Camille Petit, Imperial College London

To advance the discovery and deployment of adsorbents and adsorption-based technologies for DAC, two main research challenges must be addressed. One challenge is collecting the necessary equilibrium and kinetic data needed to assess existing and emerging adsorbents in process models for adsorbents screening. Another challenge is the creation of process-scale models that allow the evaluation and optimisation of adsorbents and adsorption technologies. In this talk, we will discuss our recent work done to tackle the former challenge. We will report the chemical, textural and thermal properties of two DAC chemisorbents as well as their equilibrium sorption properties for CO_2, N_2, H_2O, Ar and O_2 isotherms. We will also present an experimental framework we developed to characterise the internal sorption dynamics within a single adsorbent pellet. We will demonstrate its applicability to CO_2 sorption on a range of physisorbents and describe our plans to extend to chemisorbents.

Countering climate change – Filtering out CO_2 from the atmosphere and making it versatile

Tobias Horn + Leon Ketscher, Airbus

Our generation faces a significant challenge – human-induced global climate change must be halted. The Direct Air Capture (DAC) technology could be a cog in the machine by filtering CO_2 out of the atmosphere. We will discuss the extent to which new absorbent materials can contribute to more efficient CO_2 filtering processes.

A Comparative Investigation of Surface Interactions of Porous Materials using Nitrogen and Argon Adsorption

Sven Helle, Brandenburgische Technische Universität Cottbus-Senftenberg

In this presentation commercially available porous materials and synthesised porous carbon materials were investigated. Through a comparative study of nitrogen and argon adsorption isotherms, the influence of surface interactions on the adsorption was assessed. It will be shown how the difference in the adsorption of these probe gasses can be used to gain valuable information about surface characteristics of these porous materials.

An easy-to-use modification of the potential theory of adsorption

Christian Teicht, Fraunhofer-Institute for Chemical Technology

Calculate any adsorption equilibrium data from just a few experimental data? What sounds too good to be true makes Dubinin's potential theory possible in some, but unfortunately not all applications. Therefore, this talk will present an easy to handle extension of Dubinin's potential theory, which allows to extend its validity to many more adsorption equilibria.
