The Beauty of New IGC Applications

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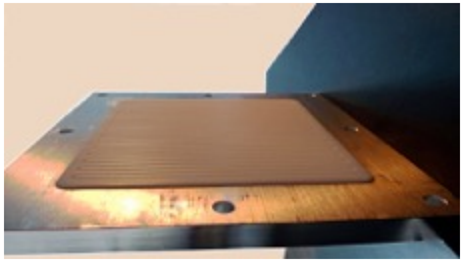
3 Examples … to inspire

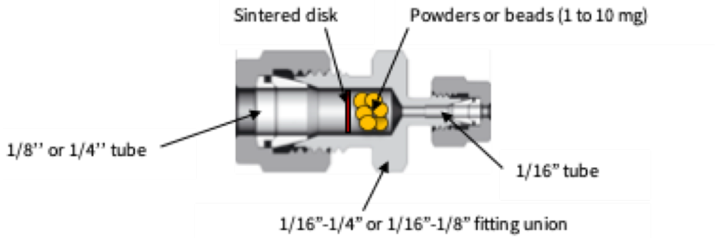
https://photoartflight.com/2016/09/21/black-swallowtail-butterfly-takeoff/

Specific surface area (BET) by water

Flat surface investigations – coatings and films



Diffusion coefficients by Zero Length Column (ZLC)



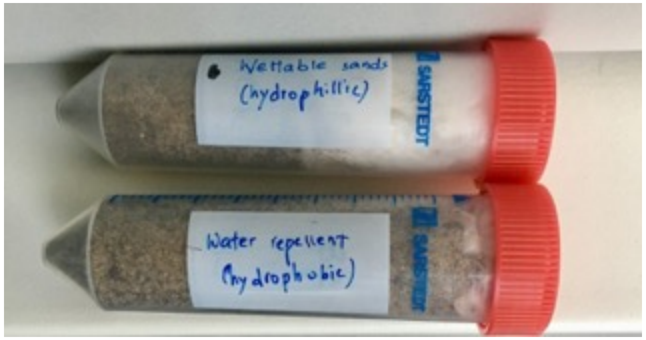
Specific surface area (BET) by water

Question
Interaction with H_2O (soils, cement)

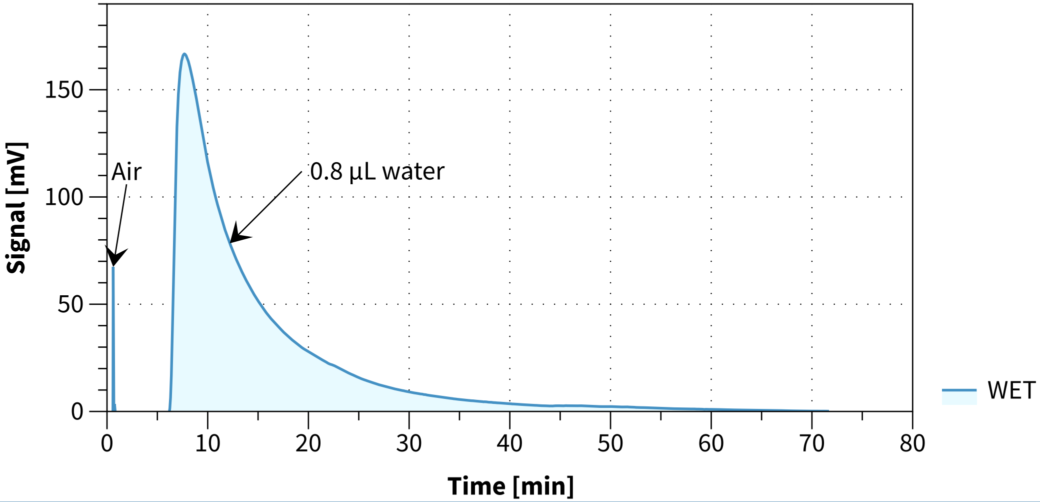
Idea / concept
H_2O as probe molecule, TCD-Detector

Results
IGC-FC: $S_{BET}(H_2O)$, heterogeneity - AEDF

Hydrophilic and hydrophobic soil (Univ. of Western Australia)



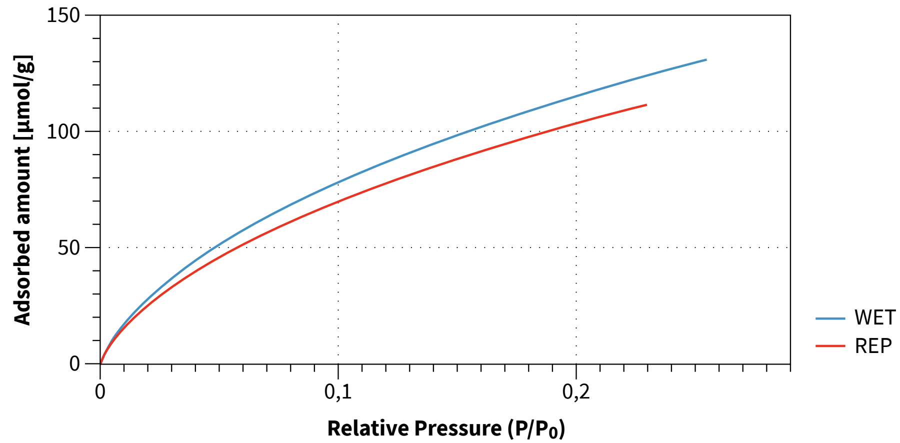
IGC-FC: saturation of sample with water-vapour and desorption curve



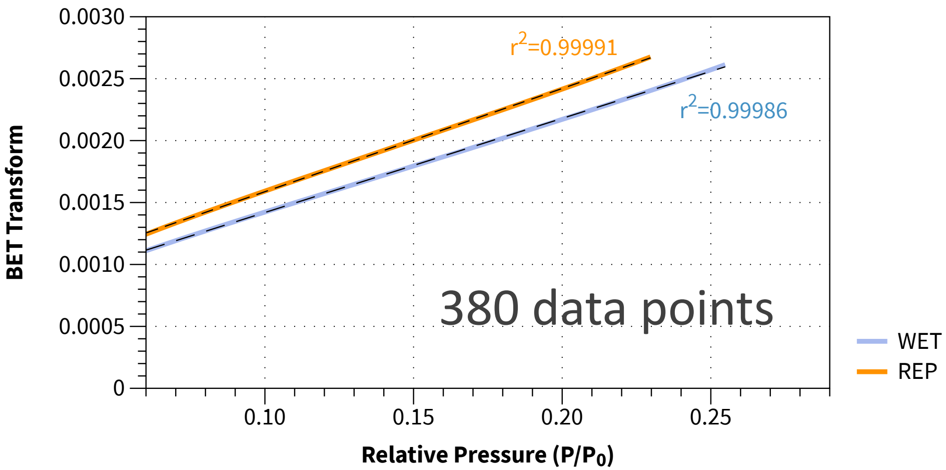
Specific surface area (BET) by water

Results
IGC-FC: $S_{BET}(H_2O)$, heterogeneity - AEDF

Calculation of adsorption/desorption isotherm: adsorbed amount vs. p/p_0



BET-regression: highly linear!



Code	Q_0 [µmol/g]	S_{BET}[m²/g]	C_{BET} [n.u.]
WET	121,4 ± 2,1	8,7 ± 0,2	11,6 ± 1,2
REP	111,5 ± 2,0	8,0 ± 0,1	12,0 ± 0,4

Specific surface area (BET) by water

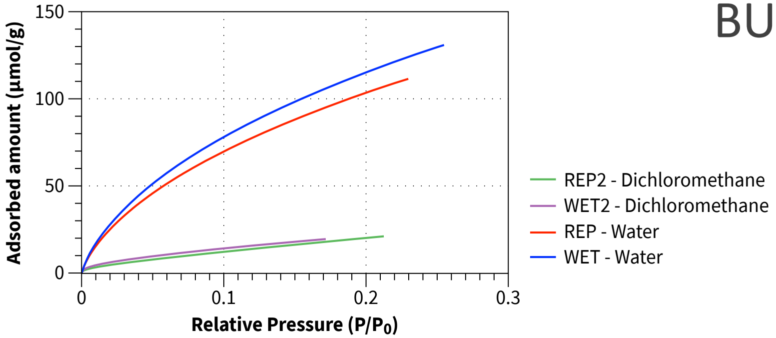
Results

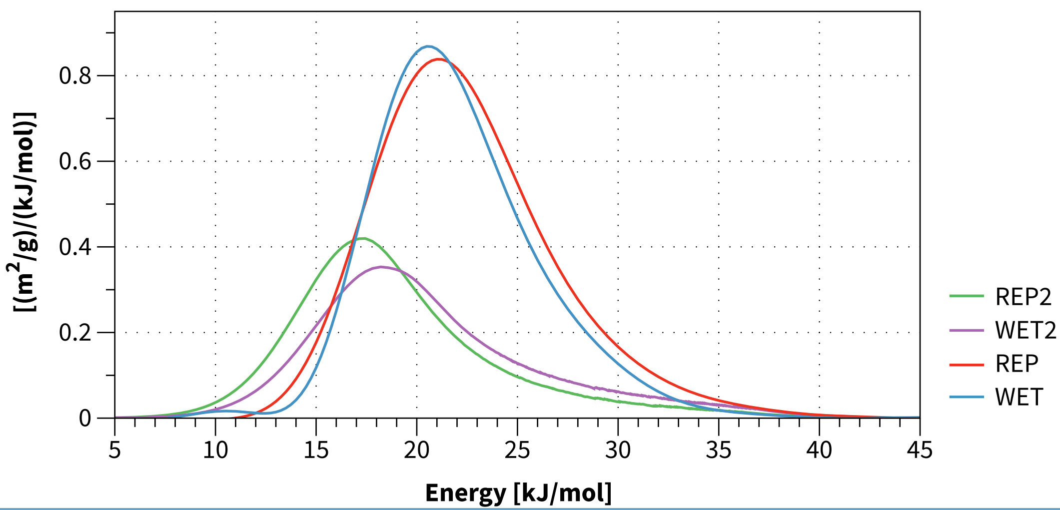
Comparison of H_2O (hydropilic) and CH_2Cl_2 (hydrophobic)

Code	Probe	Q_0 [µmol/g]	S_{BET}[m^2/g]	C_{BET} [n.u.]
WET	Water	121.4 ± 2.1	8.7 ± 0.2	11.6 ± 1.2
REP	Water	111.5 ± 2.0	8.0 ± 0.1	12.0 ± 0.4
WET2	Dichloromethane	21.6 ± 0.2	3.9 ± 0.1	13.9 ± 0.4
REP2	Dichloromethane	22.8 ± 0.2	4.1 ± 0.1	8.9 ± 0.3

Apparent specific surface area $S_{BET}(H_2O) >> S_{BET}(CH_2Cl_2)$

BUT: only small differences between wettable + repellent





Other causes of reppellency?

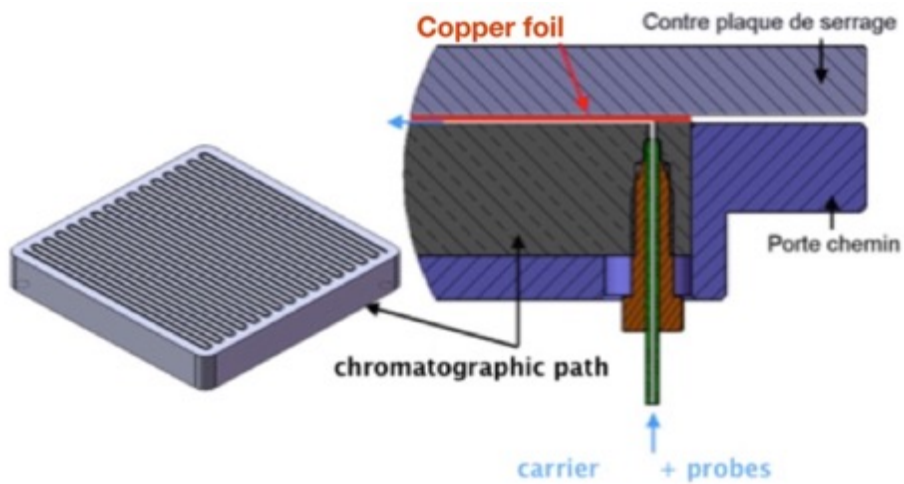
2% organic C...

Flat surface investigations – coatings and films

Question
How to measure flat surfaces? (coated metal sheets, films, polymers)

Idea / concept
use a long gas path across the surface (10x10 cm, 29 cm^2)

Results
normal IGC-ID and IGC-FC tests

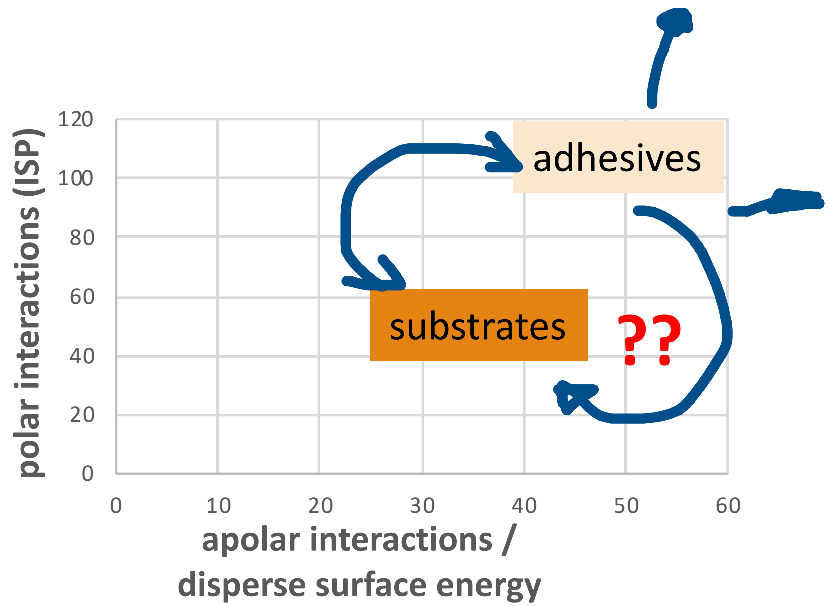


https://coleofduty.com/wp-content/uploads/2020/04/Adhesive-Film-Market.png

Flat surface investigations – coatings and films

Results
normal IGC-ID
and IGC-FC tests

- Adhesive films
  - Characterization of the polymer film
  - Characterization of the substrate
  - Compatibilities – and optimizations
  - OPEN ISSUES…



- Coated or treated metal sheets:
  - BET surface characterization, direct!
  - Surface properties
  - Use of special reagents
  - Application note (soon)

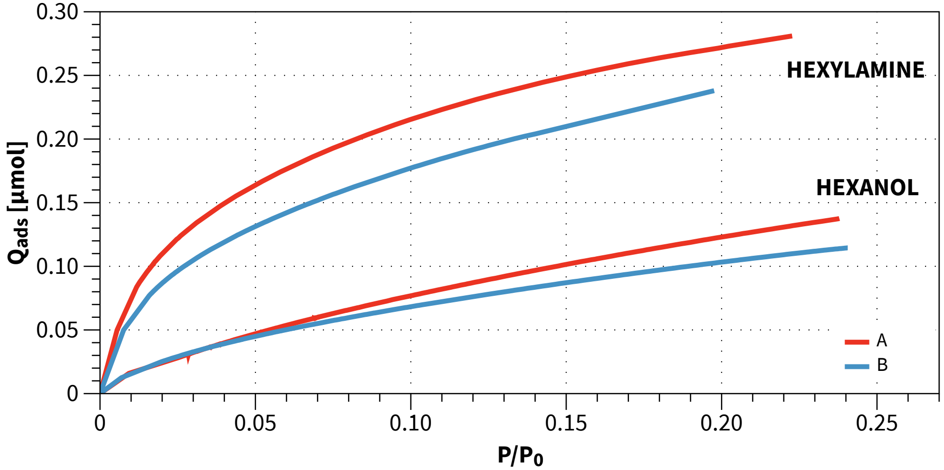
IGC on flat surfaces

Example of a steel plate degreased using two processes:

Sample A : Treatment A

Sample B : Surfactant degreasing

Objective : compare the "cleanliness" of both treatments



$S_{GEO} = 28.8\ cm^2$

		S_{BET} [m²]	S_{BET}/S_{GEO}
Hexanol	A	0.072 ± 0.016	8.0
	B	0.044 ± 0.002	4.9
Hexylamine	A	0.071 ± 0.014	7.9
	B	0.060 ± 0.005	6.7

Hexylamine and hexanol are probes for "cleanliness":

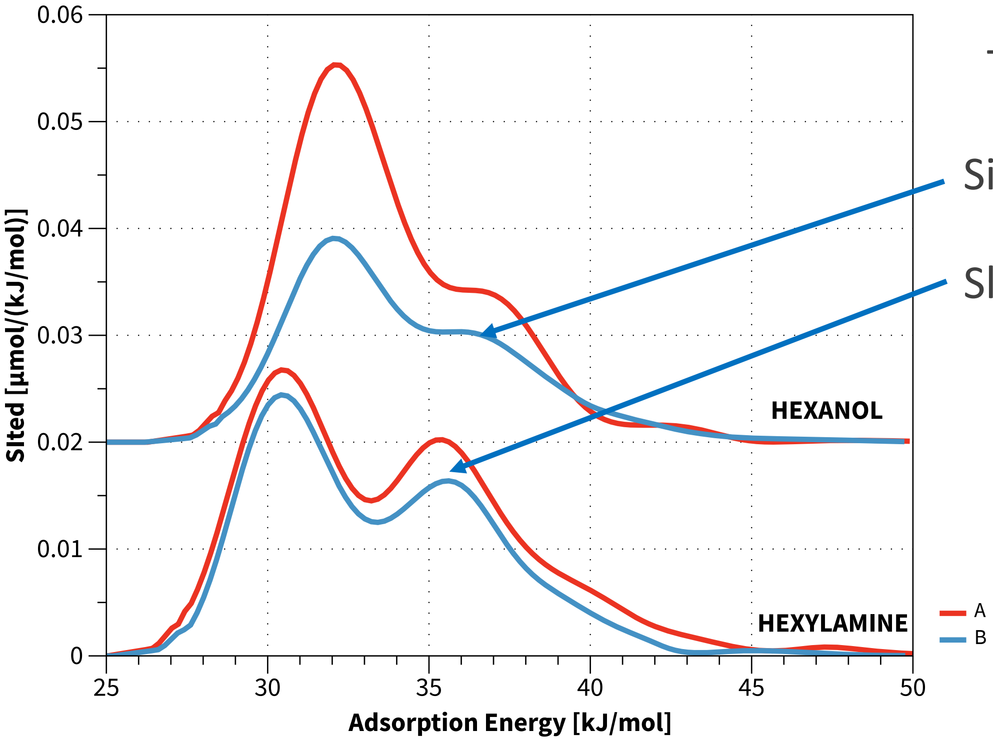
S_{BET} (A) > S_{BET}(B) => Treatment A more efficient than B

S_{BET} (hexanol) = S_{BET}(hexylamine) for A => Treatment A: more homogeneous

S_{BET} (hexanol) < S_{BET}(hexylamine) for B => Treatment B: a part of the basic sites remain inaccessible

Surface heterogeneity: even on flat surfaces

Adsorption Energy Distribution Function (AEDF)



Two kinds of sites (bimodal AEDF)

Significantly less basic sites on B

Slightly less acidic sites on B

Treatment A is more efficient

to regain adhesive strength of the steel

(especially visible with hexanol)

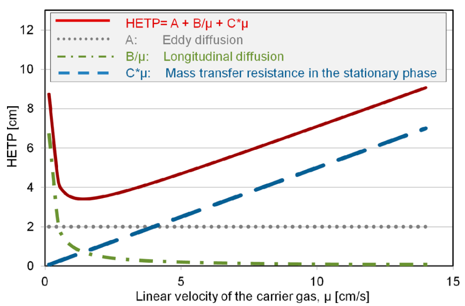
Diffusion coefficients by IGC

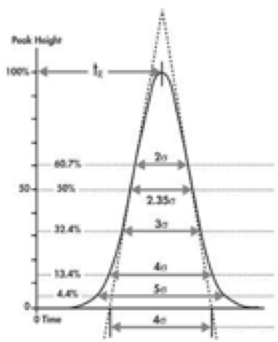
Question
How to improve current D_{eff} determination?

Idea / concept
use IGC-FC possibility and desorption curve

Results
D in porous samples by any gaseous probe

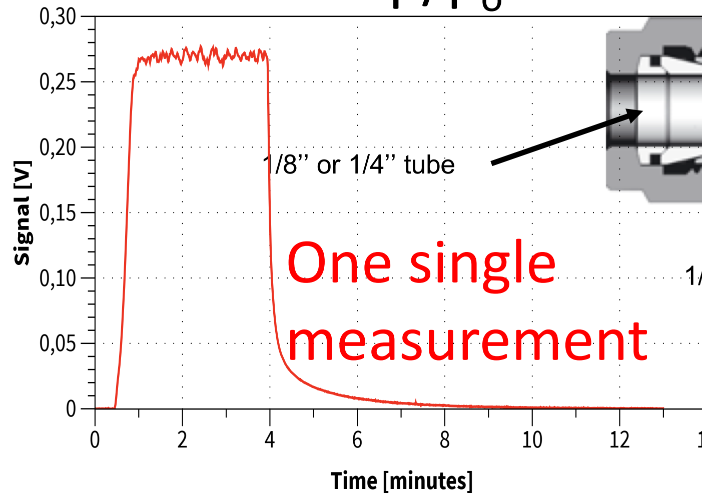
Currently: van Deemter – variation of flow rates and calculation of HETP (height equivalent to a theoretical plate) Time consuming!

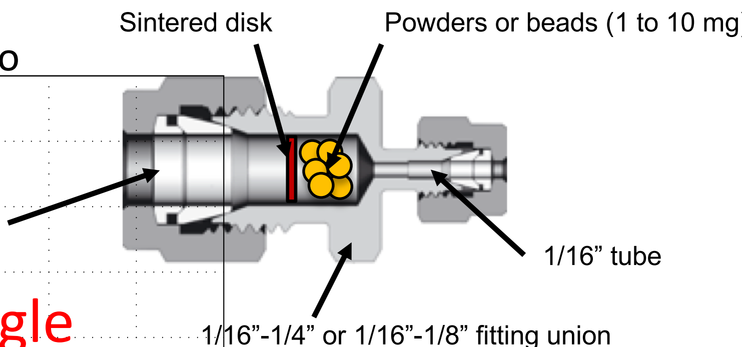




Zero Length Column (ZLC)

Saturation at p/p_o





One single measurement

Diffusion coefficients

Results

D_{eff} in porous samples by any gaseous probe

ZLC method:

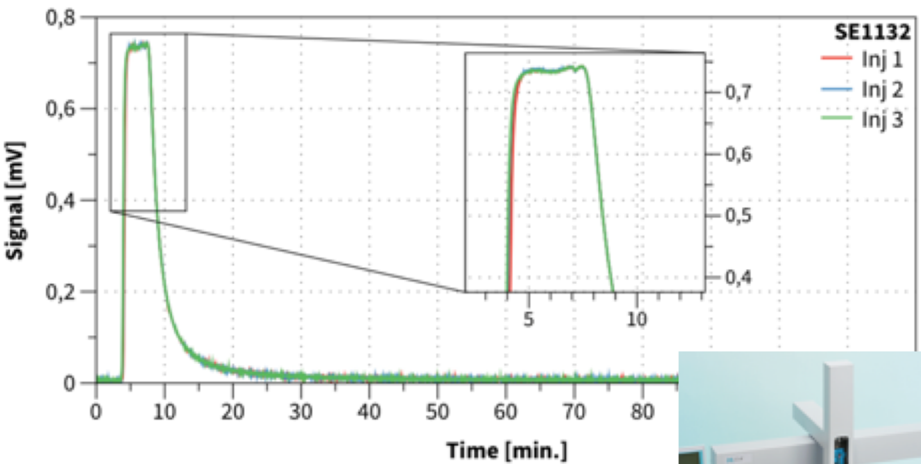
Conversion of the desorption front and fitting the asymptote in a Ln(C/C0) plot to obtain D.

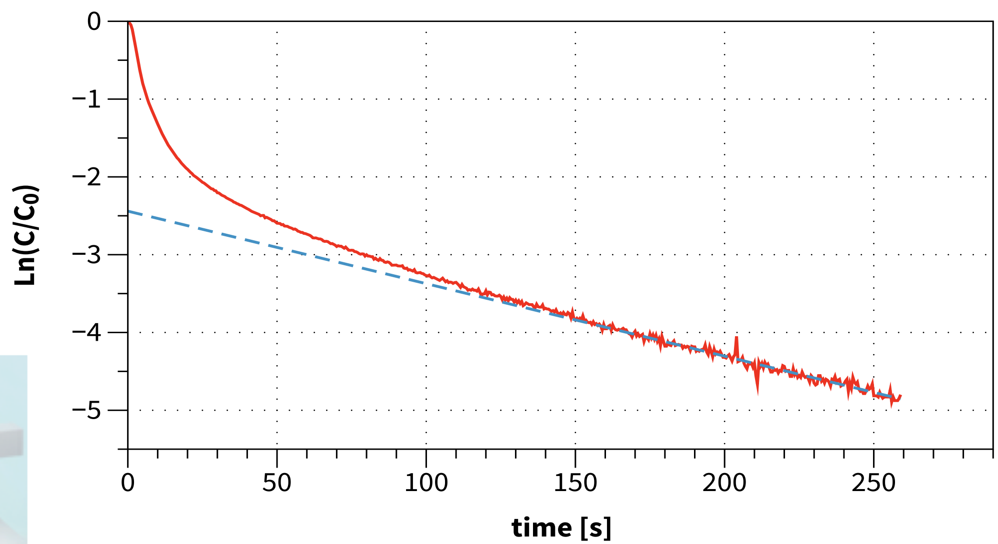
$$Ln\left(\frac{C}{C_0}\right) = Ln\left(\frac{2L}{\beta^2 + L(L-1)}\right) - \beta^2 \frac{D.t}{R^2}$$

References

M. Eic, D.M. Ruthven, Zeolites, 8(1), 40–45, (1988)

D. Ruthven, F. Brandani, Adsorption, 11(1), 31–34, (2005)





Example: Diffusion coefficients

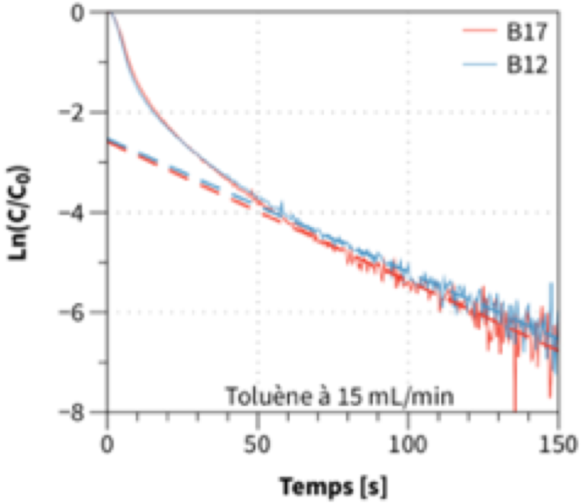
Example: single Alumina beads

B12: d_{50}= 1.71 mm

B17: d_{50}= 1.65 mm

$D_{toluene} < D_{cyclooctane} < D_{octane}$

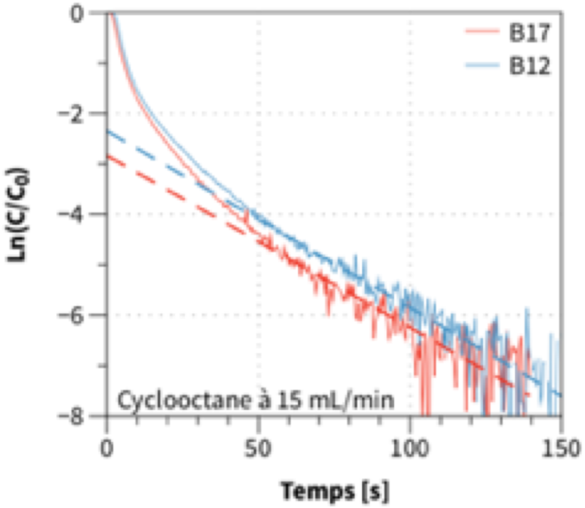
toluene



D_{tol}= 2.14*10^{-5} cm^2/s

D_{tol}= 2.07*10^{-5} cm^2/s

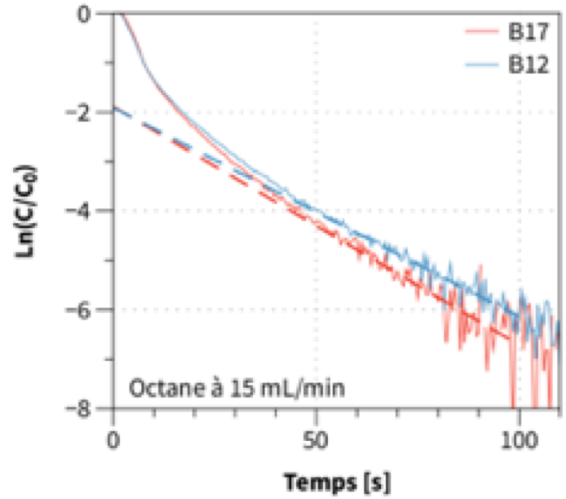
cyclooctane



D_{cyc}= 2.85*10^{-5} cm^2/s

D_{cyc}= 2.50*10^{-5} cm^2/s

n-octane



D_{oct}= 3.63*10^{-5} cm^2/s

D_{oct}= 3.87*10^{-5} cm^2/s

Beauty of many possibilities

- Flexible use of (almost) any vaporizable probe
  - Custom-oriented tasks: corrosive gases, functional groups (double bonds…)
- Saturation at p/p_0 by constant flow with any probe (vaporizable)
- Specific surface area measurements with any probe
- with 22+ probes: good coverage of 3-dimensional HSP sphere
  - HSP: Hansen Solubility Parameters for formulations
- Detectors for organics (FID) and inorganics (TCD)
- Device for flat surfaces

How would you like
to characterize your samples
with 15+ gas probes?

Take your opportunities ...

... and use your imagination!

Demands, ideas or specific questions?

Don’t hesitate to ask!

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