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Simposio sobre Adsorción Adsorbentes y sus Aplicaciones

DEQ-UFC
15 Anos

ADSORCIÓN DE GASES APLICADA A PROCESOS DE ALMACENAMIENTO Y SEPARACIÓN

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Departamento de Engenharia Química
Grupo de Pesquisa em Separações por Adsorção

San Luis, 25 de Febrero de 2009

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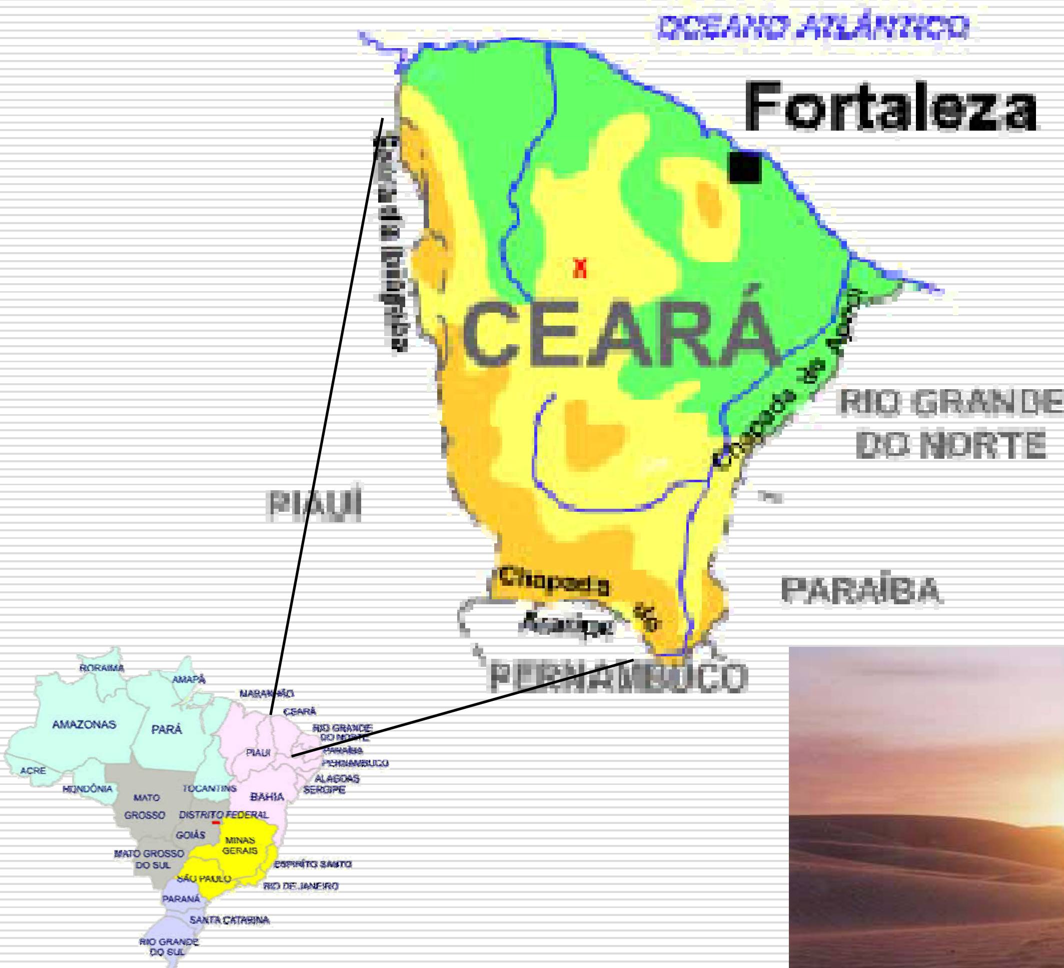
Brasil – Ceará – Fortaleza – UFC



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Nuevas tecnologías para un medio ambiente más limpio

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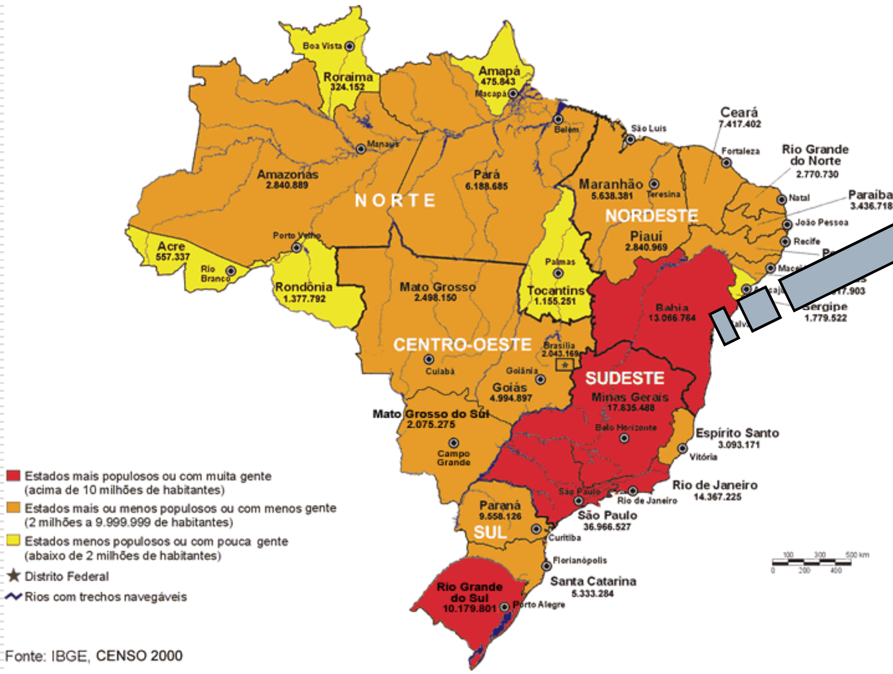
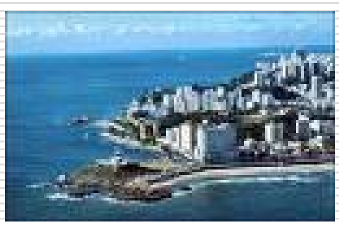


Salvador, Bahia
15 a 19 de júnio de 2009
Curso de formación complementária de la UNIA
www.unia.es

TEMAS

- El hidrógeno como combustible alternativo.
- Nanotecnologías y medio ambiente
- Control catalítico de emisiones de NOx
- Nuevos materiales para la obtención de combustibles via Fischer-Tropsch
- Biocombustibles; situación actual y perspectivas



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Outline

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Adsorption for storage purposes – the case of natural gas

- ✓ Activated carbon, the unbeatable sorbent
- ✓ Measurement of adsorption equilibrium (methods, pros and cons, data)
- ✓ Charge and discharge cycles (long term performance)
- ✓ Multicomponent adsorption measurements

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- ✓ Column dynamics and PSA processes

Concluding remarks

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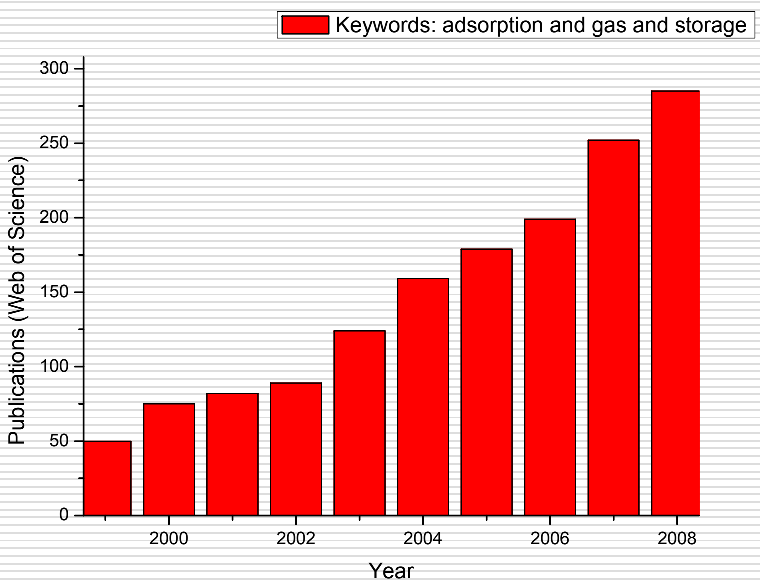
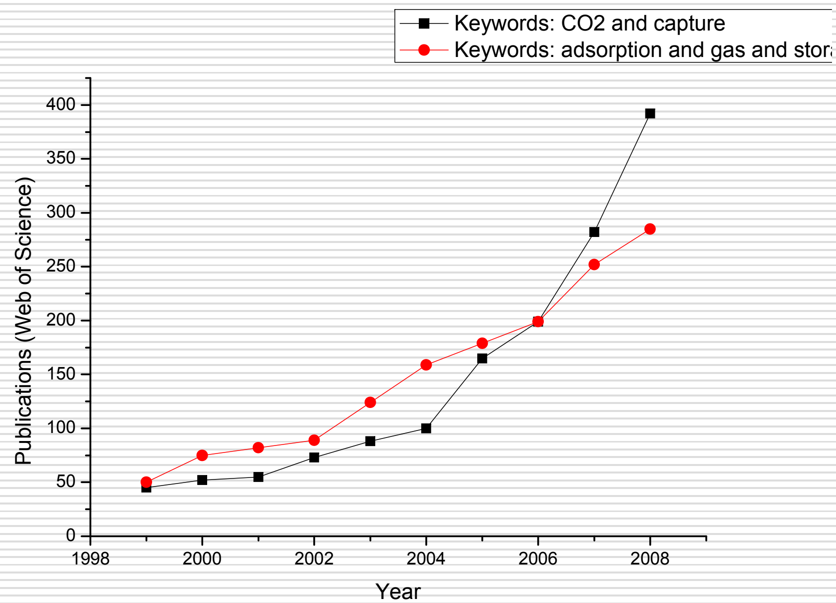
Evolution of publications
(Web of Science)





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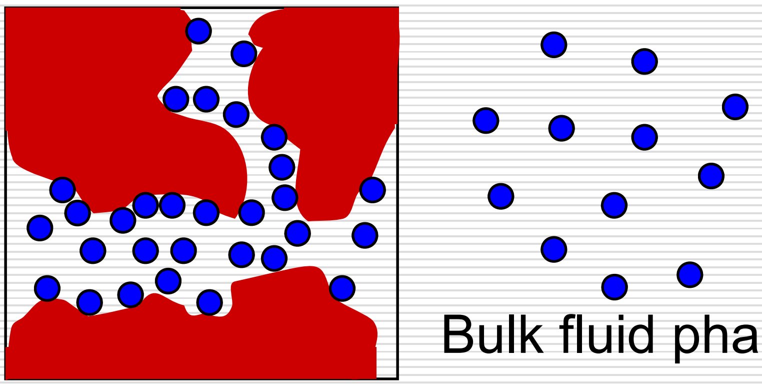
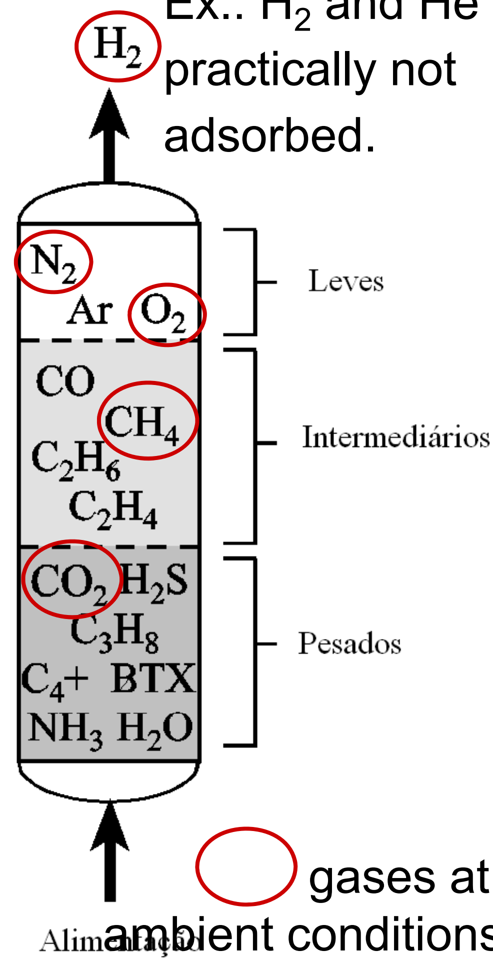
Principles of adsorption

Adsorbed
phase



Bulk fluid phase

Ex.: H_2 and He are practically not adsorbed.



gases at ambient conditions

Important factors:

- *Volatility (T_c)*
- *Polarity (dipole/quadrupole moment)*
- *Sorbent surface area / pore volume / PSD / surface chemistry*

Excess Adsorbed Phase Concentration, m_{ex}

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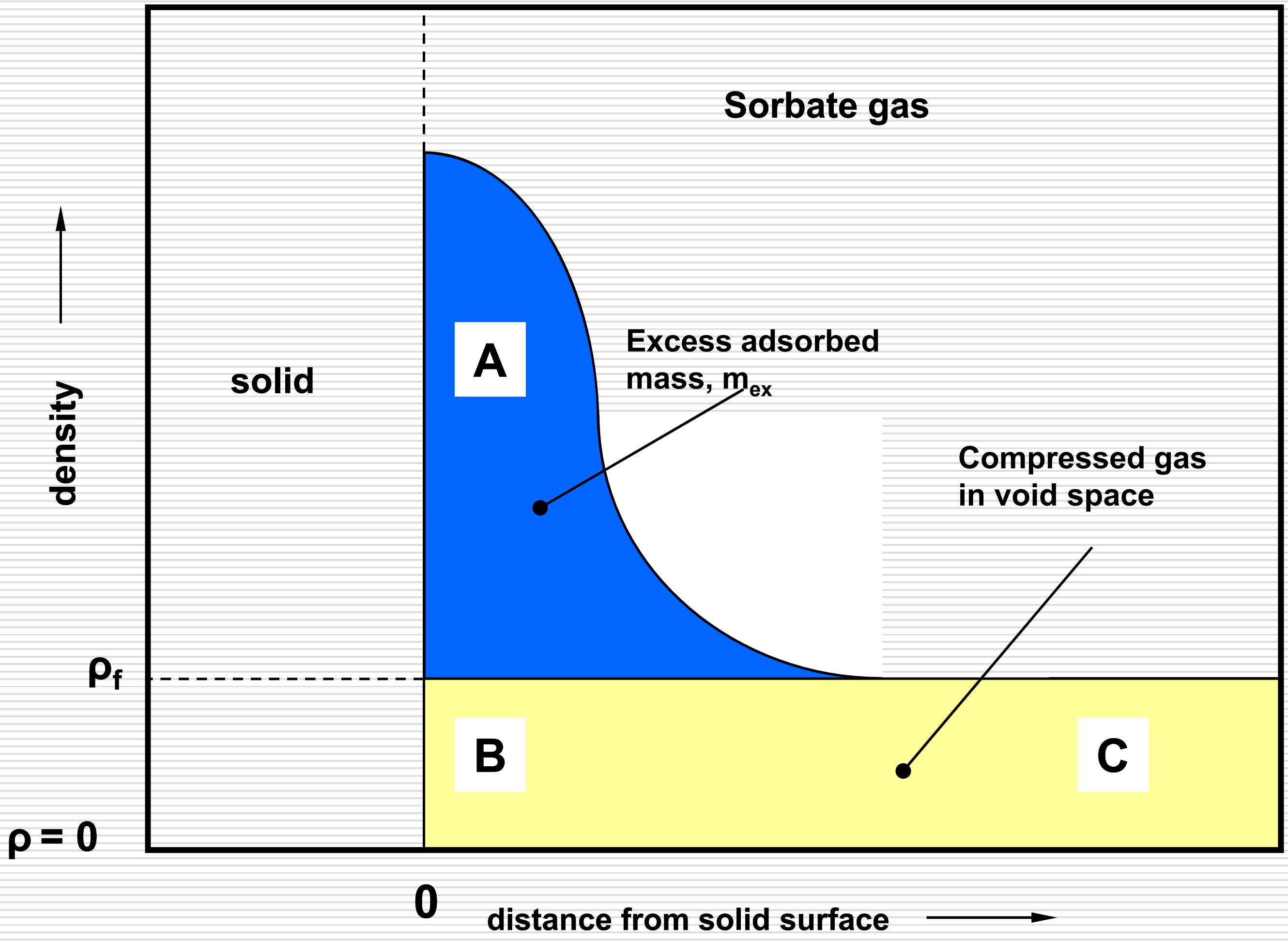

Absolute Adsorbed Phase Concentration, m_{abs}

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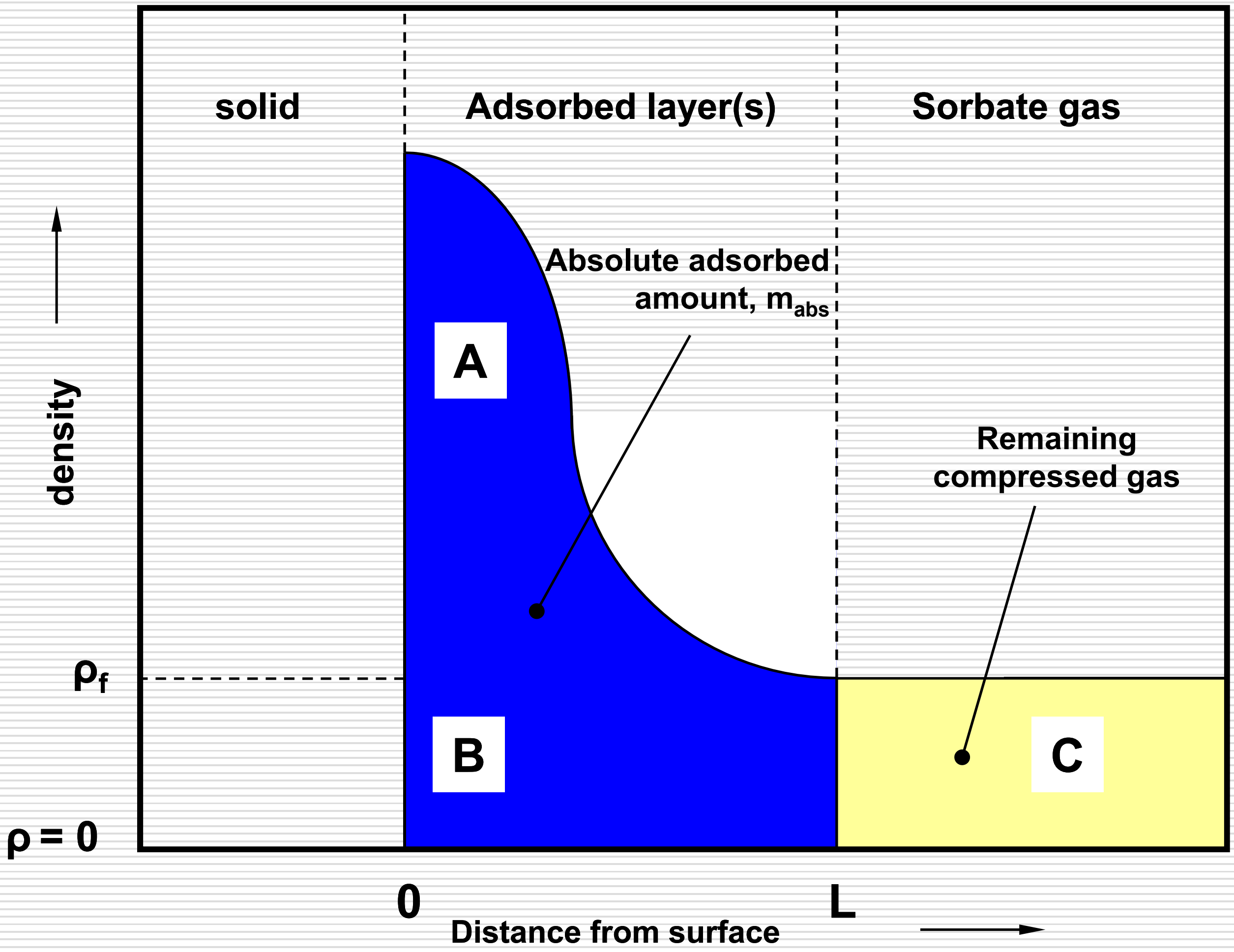

- Dreisbach et al., Adsorption 8, 95, 2002.
- Do and Do, Carbon 41, 1777, 2003
- Bastos-Neto et al., Adsorption 11, 911, 2005

For vapors,
$\rho_f << \rho_{ads}$ ($\sim\rho_{liq}$)
Hence $m_{ex} \sim m_{abs}$

For gases,
ρ_f is comparable to ρ_{ads}
Hence $m_{ex} < m_{abs}$

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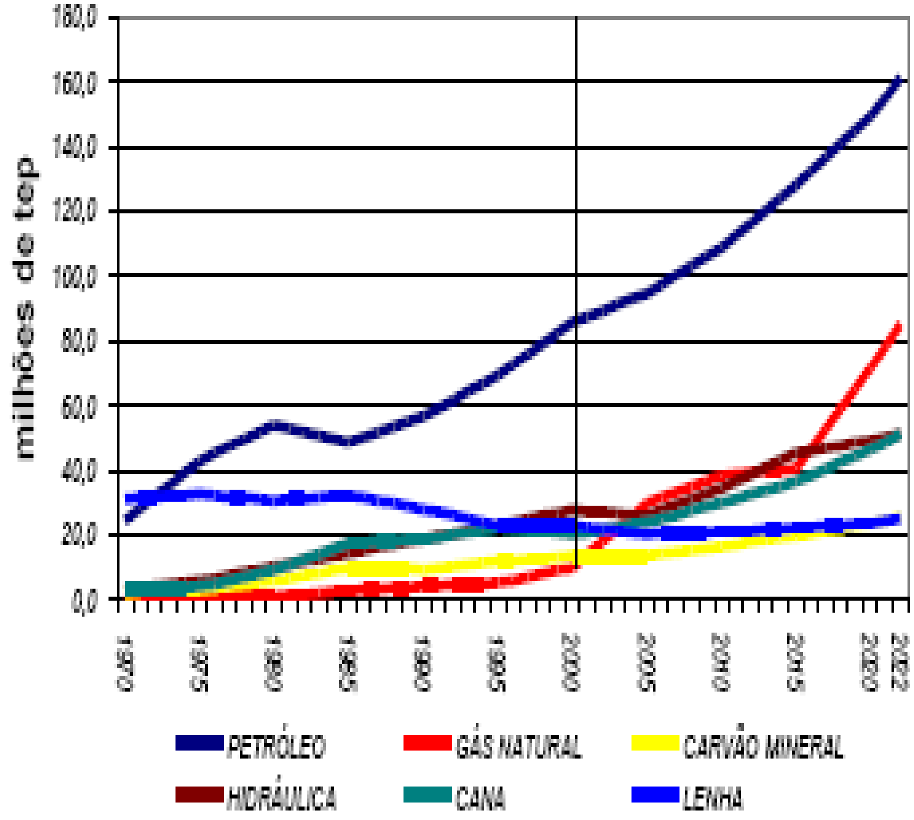
Why Store Natural Gas in Brazil?

ENERGY SOURCES (%)

	2000	2020
Petroleum Derivatives	43	40
Hydro	30	16
Ethanol/Bagasse	11	12
Wood	8	6
Coal	5	6
Natural Gas	3	20



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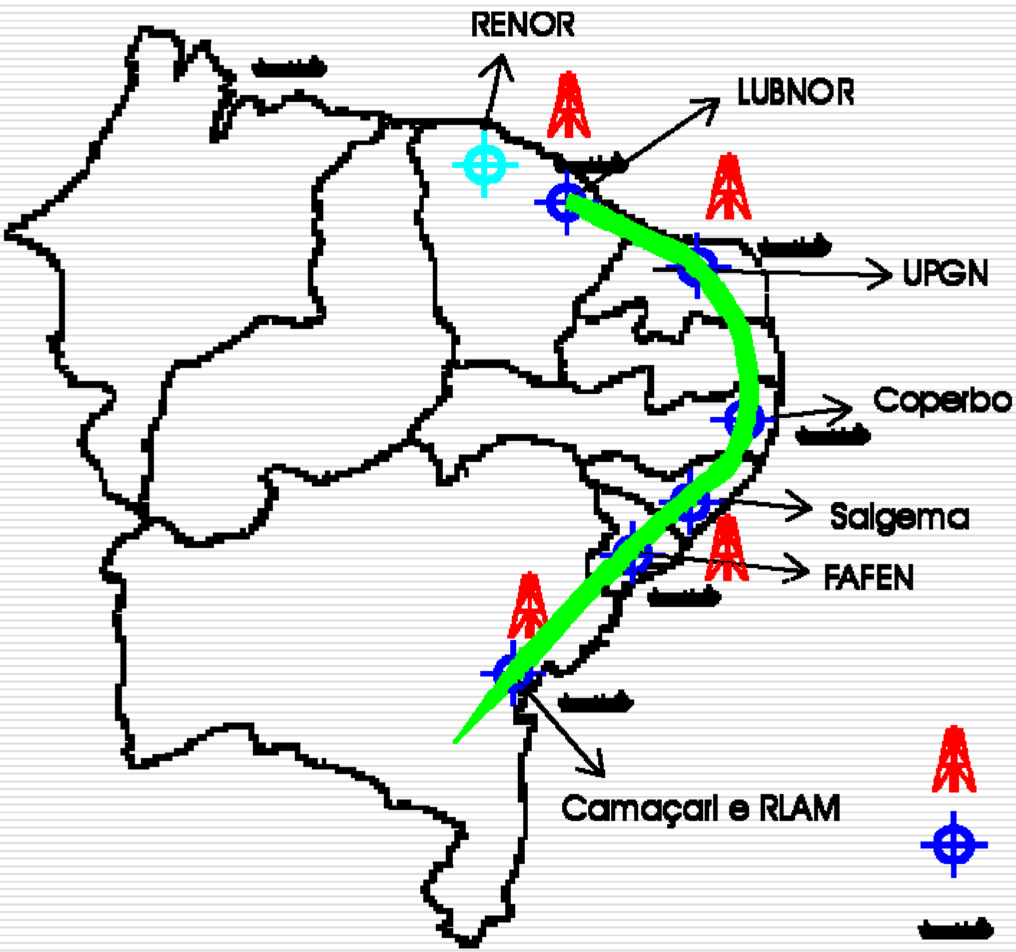
Why Store Natural Gas in Brazil?



"Mobile pipelines" alternatives:

	Vol Ratio (Gas/Vessel)
➢ Liquified (LNG):	600
➢ Compressed (CNG):	200
➢ Adsorbed (ANG): Today	60-100
DOE Target	150 (180)

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What to look for in an adsorbent for gas storage?

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- High capacity
- Selectivity for the target stored molecule
- Irreversibility/regenerability
- Low cost
- Chemical inertness
- High bulk density (if stored volume is an issue)
- Mechanical resistance

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Activated carbon: the unbeatable sorbent for NG storage

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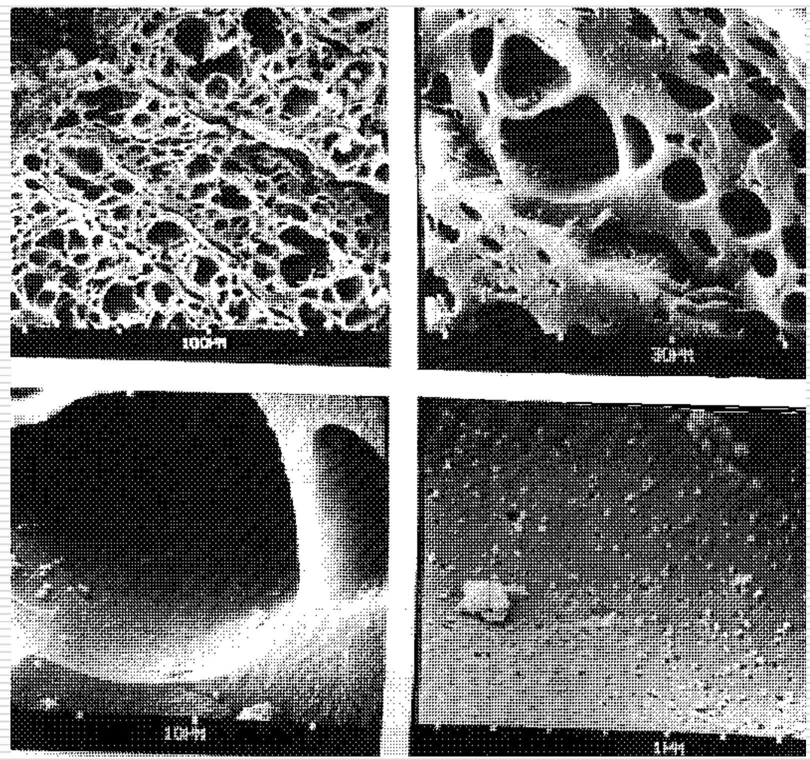
- Hydrophobic character, affinity for organic molecules
- Inexpensive raw material (agroindustrial/lignocellulosic wastes)
- Possibility of tailoring surface area and PSD according to activation procedures



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Coconut shell-based activated carbons

Sample	Activation Agent	Pirolysis Conditions	BET area (m^2/g)	Micropore Volume (cm^3/g)	Stored Methane (V/V)	
Sutcliffe (SRD-21)	_	_	1967	0.945	105	
Mead Westvaco	-	-	1906	0.984	80	
CA29W2 (air)	H_3PO_4	450/1/2:00	727	0.384	--	
CA36W2 (air)	H_3PO_4	450/1/2:00	844	0.502	95	
CA44W2 (air)	H_3PO_4	450/1/2:00	767	0.440	--	[2] [1]
CA51W2 (air)	H_3PO_4	450/1/2:00	779	0.441	59	
CA51W2N	H_3PO_4	450/1/2:00	1441	0.729	82	
CAQF-30	$ZnCl_2$	500/4/3:00	2114	1.142	82	
CAQ-26	$ZnCl_2$	500/4/3:00	1048	0.705	--	
CAQ-29	$ZnCl_2$	500/4/3:00	1266	0.628	--	
CAQF-P (0.09)	H_3PO_4	850/10/2:20	--	~0.600	85	[3]
CAQF-Zn (0.25)	$ZnCl_2$	850/10/2:20	--	~ 0.55	86	

[1] Azevedo et al. Microporous and Mesoporous Materials 99 (2007) 360.
[2] Rios et al. Adsorption (2009) accepted.
[3] Prauchner and Rodríguez-Reinoso Microporous and Mesoporous Materials 109 (2008) 581

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Which factors determine adsorption capacity?

Although it is accepted that adsorption capacity is proportional to surface area,

Carbon sample	BET Surface Area ($m^2.g^{-1}$)	Average Pore Width – HK (Å)	Total Pore Volume ($cm^3.g^{-1}$)	Micropore Volume - DR ($cm^3.g^{-1}$)	Micropore Volume - MC ($cm^3.g^{-1}$)
C1	1822	15.0	1.114	0.749	0.988
C2	1522	12.5	0.957	0.674	0.850
C3	1261	13.0	0.775	0.550	0.795
C4	1438	12.3	0.924	0.761	0.953
C5	1668	11.1	1.041	0.625	0.901
C6	2021	14.5	1.299	1.123	1.166
C7	1512	12.2	0.873	0.793	0.793
C8	1301	13.8	0.775	0.712	0.461
C9	1052	13.7	0.622	0.576	0.603
C10	2336	13.1	1.431	0.958	1.296

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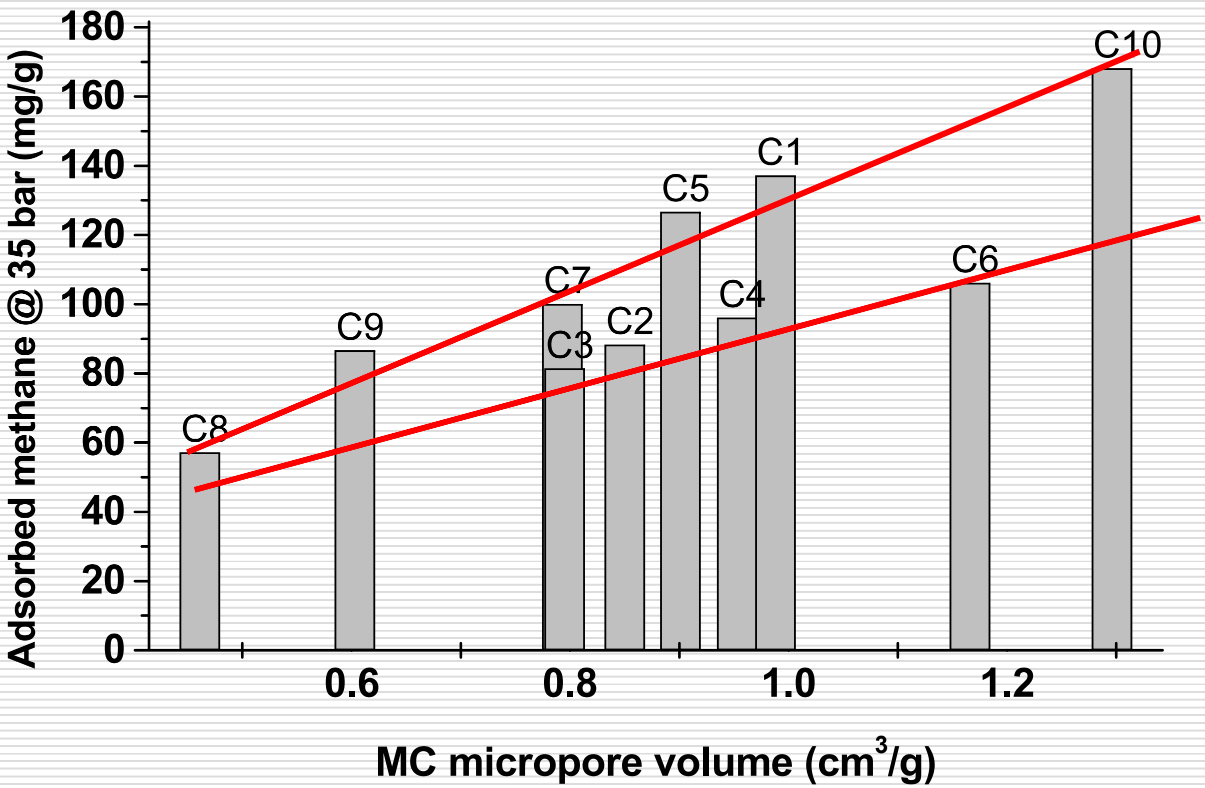
Which factors determine adsorption capacity?



Bastos Neto et al. Applied Surface Science 253 (2007) 721.

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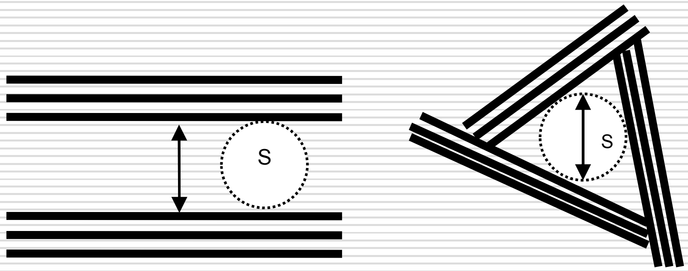
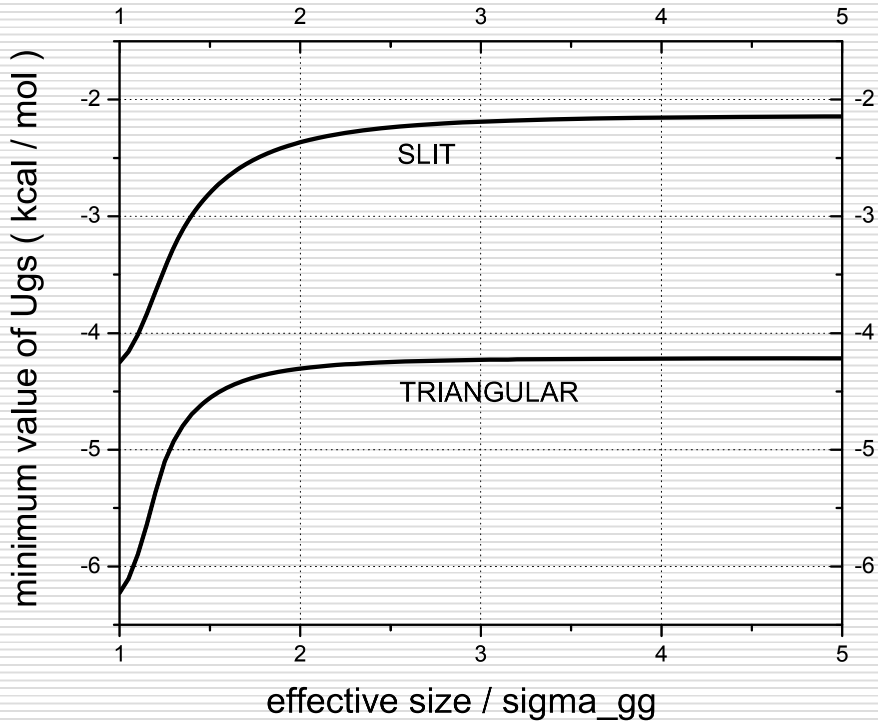
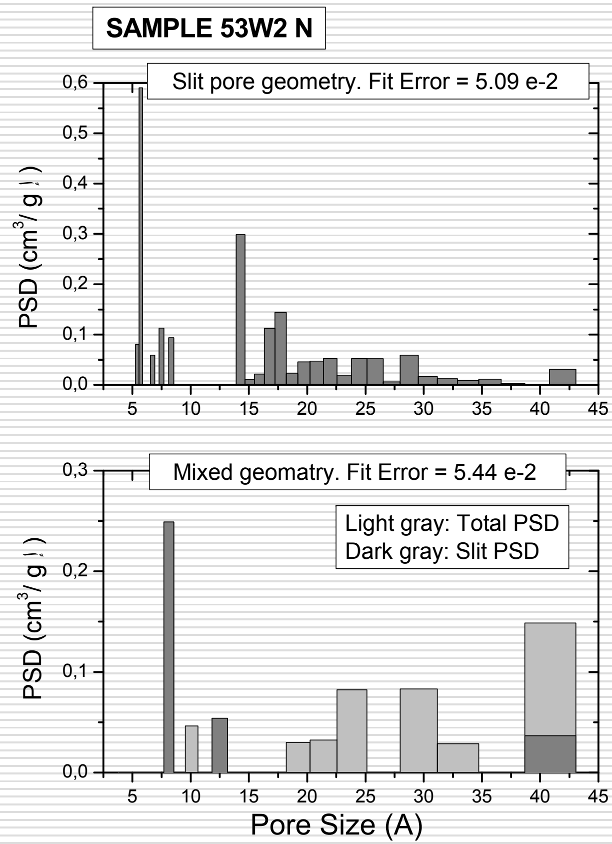
Are all pores of an activated carbon really slit-like?







AZEVEDO *et al.* Journal of Materials Chemistry 2009 (in press)

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Concluding remarks

Measurement of Adsorption Equilibrium Data

Pure Gas Isotherm

- Gravimetry
- Volumetry
- Break through curves

Mixed Gas Isotherm

- Volume-Gravimetry
- Volumetry with Gas chromatography (GC)
- Modified van Ness Method
- Sum-Isotherm-Method

Gravimetry

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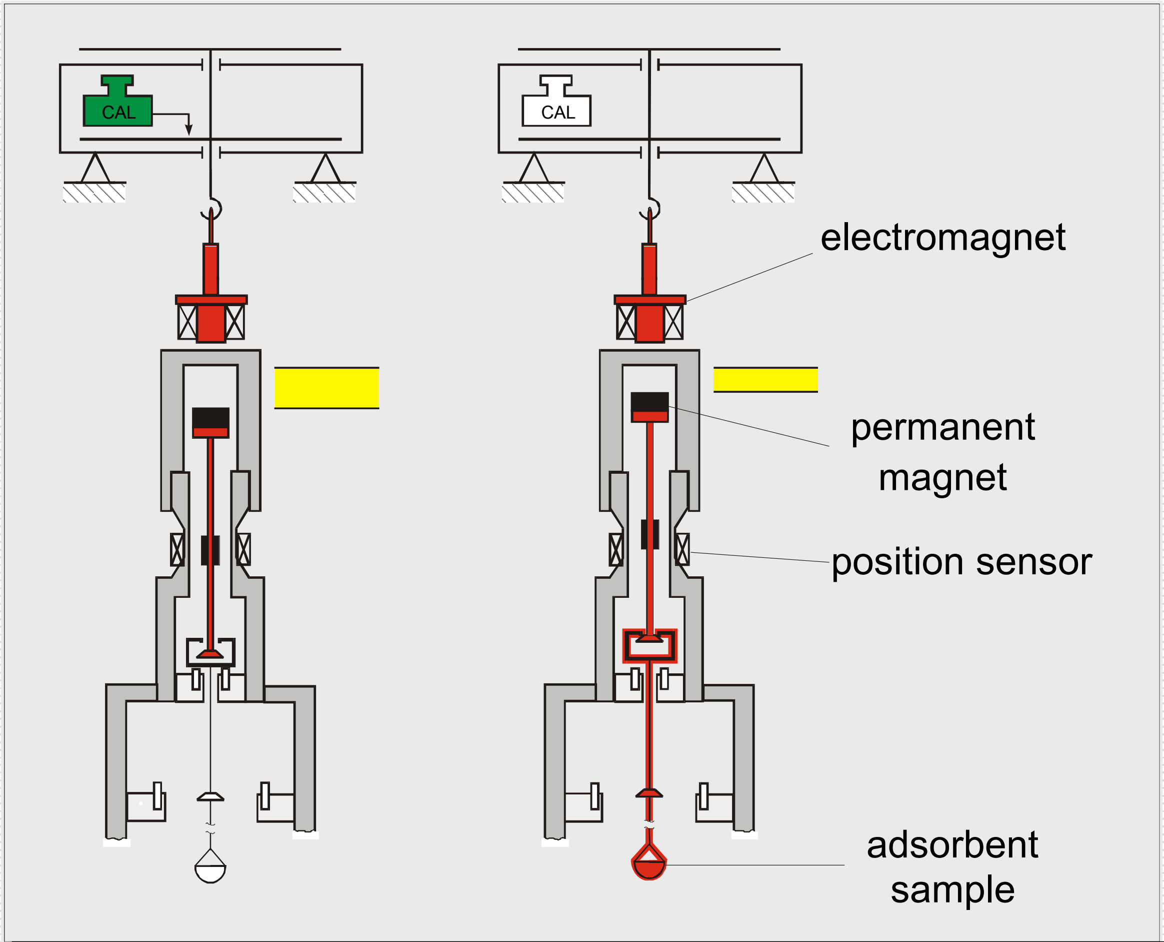

Calibration of instrument:
sample holder and solid sample specific volume

Measurement:
***p*, *T*, m_{MB}**

Calculation:

$$m_{ex}(p,T) = m_{MB}(p,T) + (\overline{V}_b + \overline{V}_s)\cdot \rho_f(p,T)$$

m_{ex} is the ACTUALLY and ACCURATELY measured quantity !!!

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Volumetry

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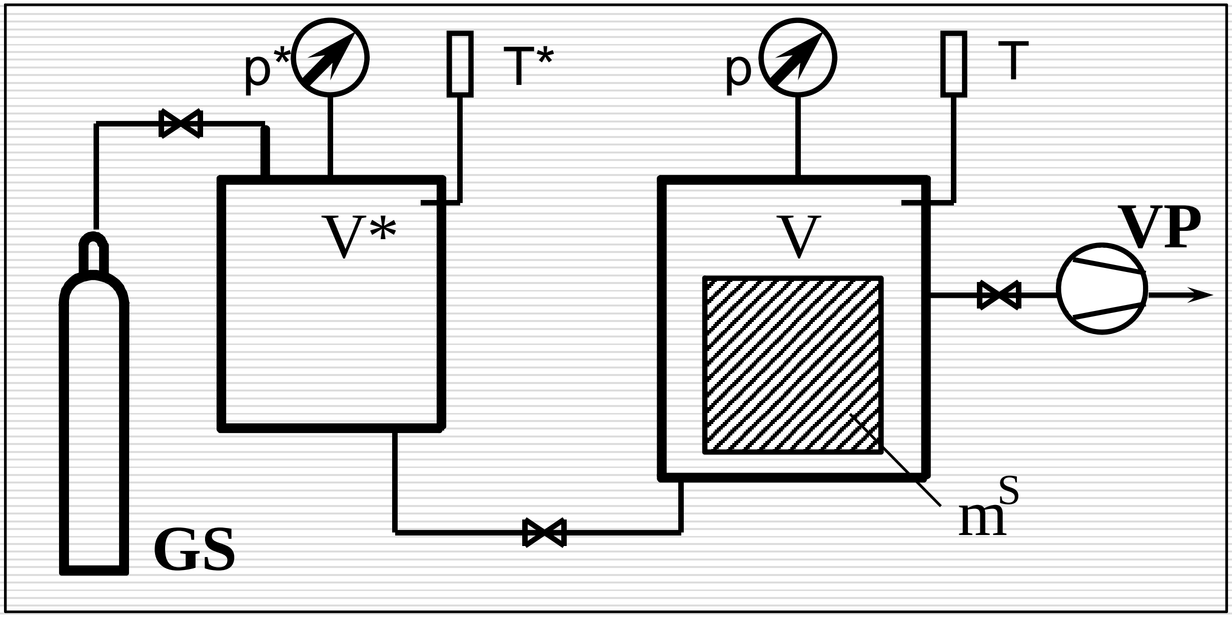

Calibration of instrument: $m_{ex} = m_{abs} - V^{ads} \cdot \rho_f(p,T)$

Volume of vessel ... $= V^* \cdot \rho_f(p^*,T^*) - (V^*+V) \cdot \rho_f(p,T)$

Measurement: p, T

m_{ex} is the ACTUALLY and ACCURATELY measured quantity !!!

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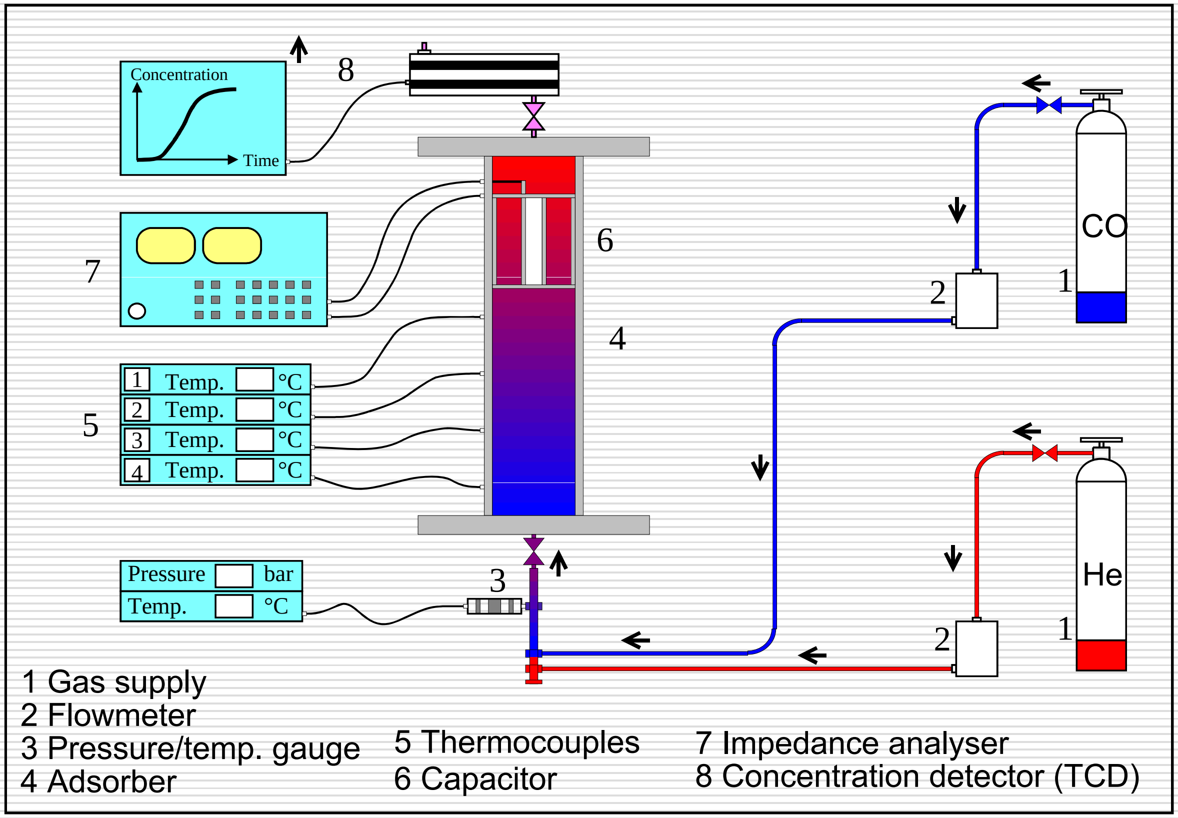
Breakthrough curves



Calibration of instrument: bulk density ...

Measurement: concentration c(t), massflow m_{flow} time t

Calculation:

$$m_{ex} = m_{abs} - V_{ads}\rho_f$$

$$= \dot{m}_{flow} \times t - V_{col} \times \rho_f$$

m_{ex} is the ACTUALLY and ACCURATELY measured quantity !!!

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Measurement of Adsorption Equilibrium Data – pros and cons

Gravimetry

- Direct measurement of m, p, T
- Mass change during sample preparation
- Uptake curve
- Adsorption isotherm, (Kinetics)

Volumetry

- Direct measurement of p, T
- "Simple" apparatus
- Adsorption isotherm

Breakthrough curve

- Direct measurement of c, p, T
- "Simple" apparatus
- Concentration dependency in carrier gas
- Close to technical separation

Gravimetry	Volumetry	Breakthrough	Gravimetry dyn.
$\Delta m/m$ = 0.1 %	$\Delta m/m$ = 0.5 %	$\Delta m/m$ = 0.5 %	$\Delta m/m$ = 0.25 %

R. Staudt, Proceedings of FOA 8, Sedona, USA, 2004

Adsorption isotherms for selected gases at high pressures

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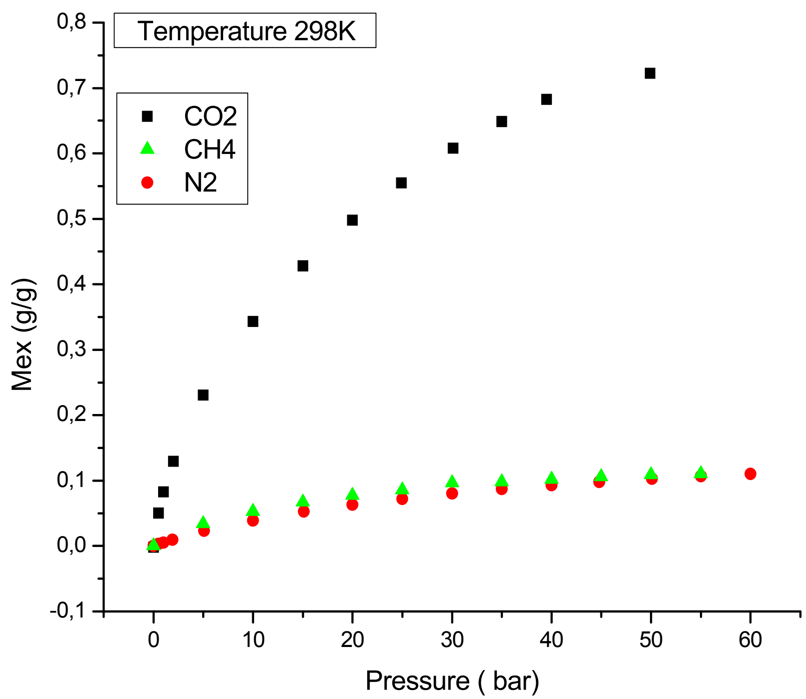
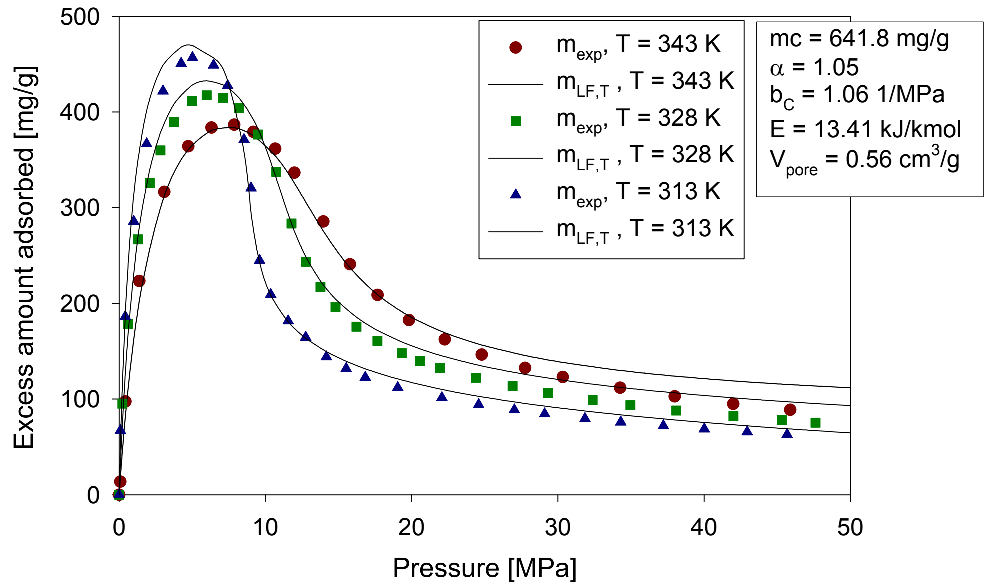


Activated carbon Mead Westvaco WV 1050



CO_2 on activated carbon Norit R1 Extra

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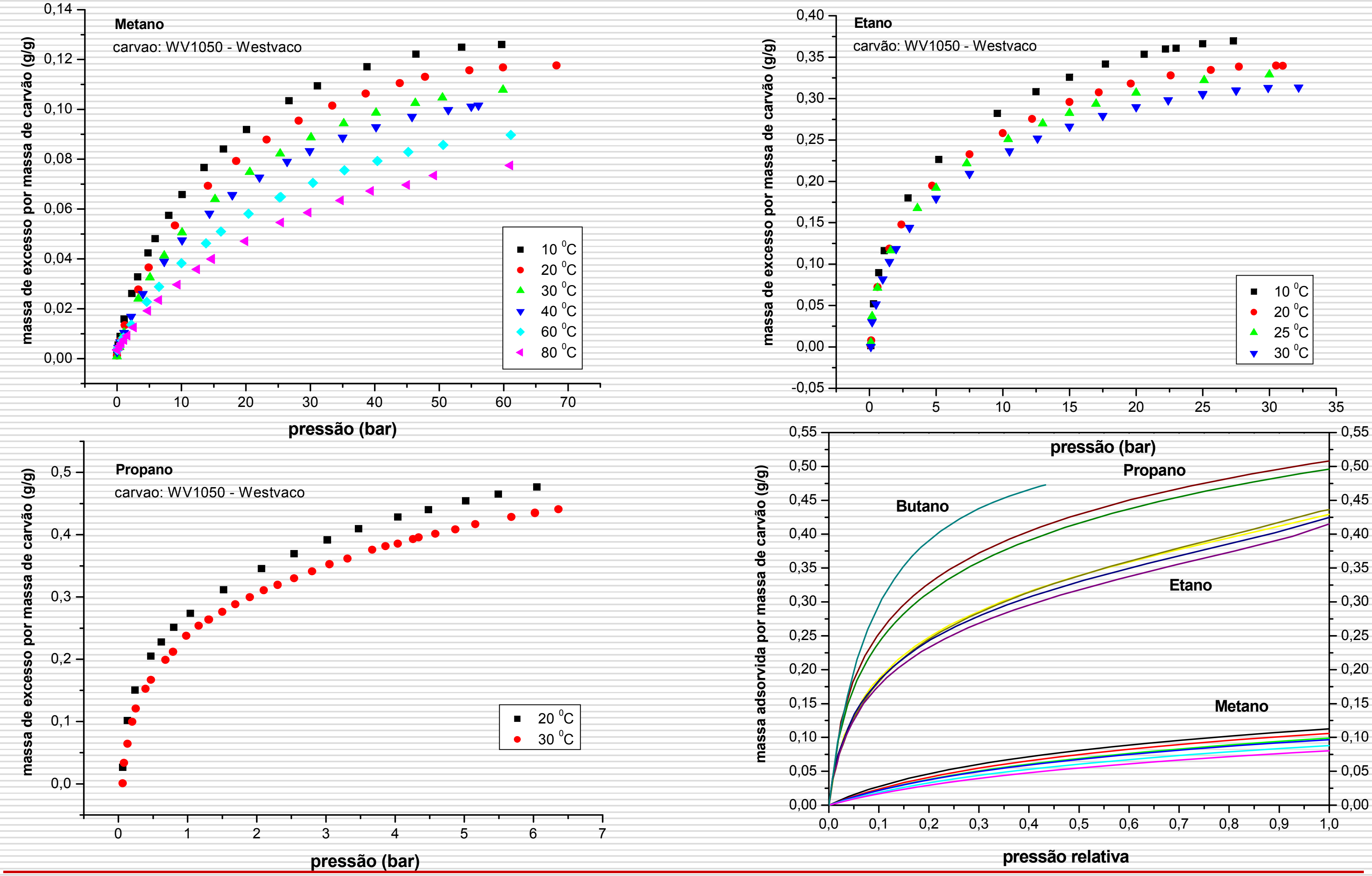
Adsorption isotherms for natural gas components

Amora et al., LAAR 2009 (in press)



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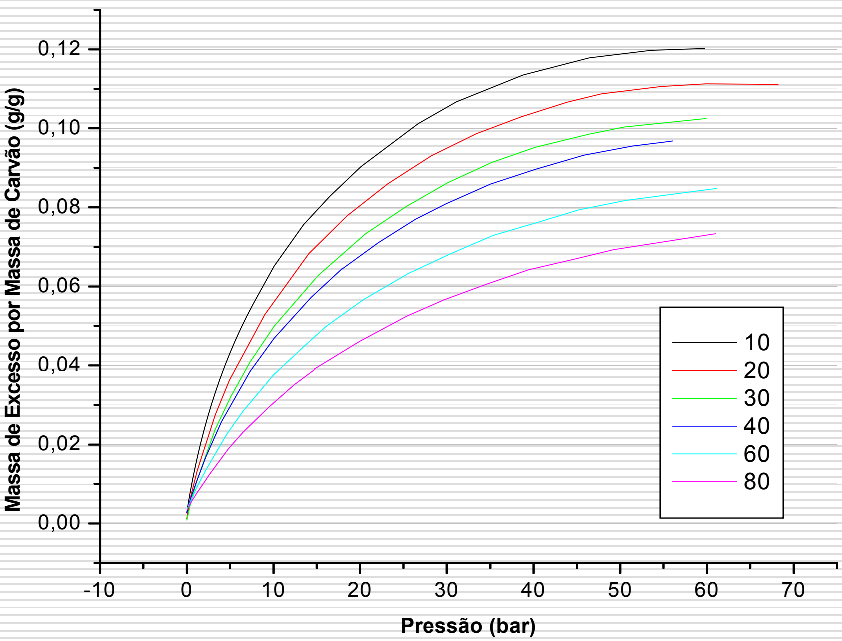
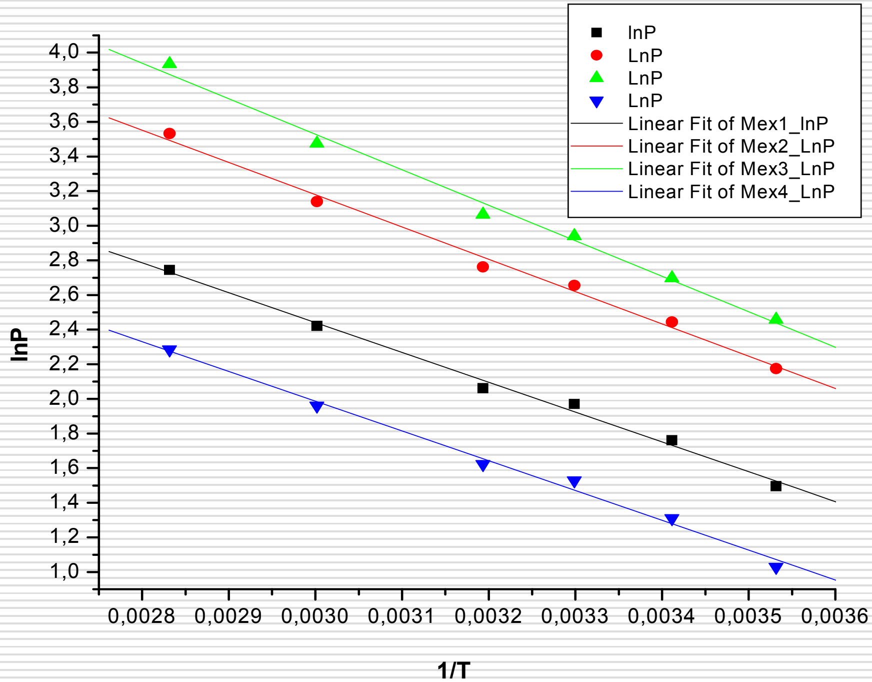
Determination of isosteric heat of adsorption





$$\left(\frac{\partial}{\partial T}\ln[p]\right)_{m_{ex}} = -\frac{\Delta H_{Ads}}{RT^2}$$

$$\Delta H_{Ads} = R \cdot \left(\frac{\partial}{\partial(1/T)}\ln[p]\right)_{m_{ex}}$$

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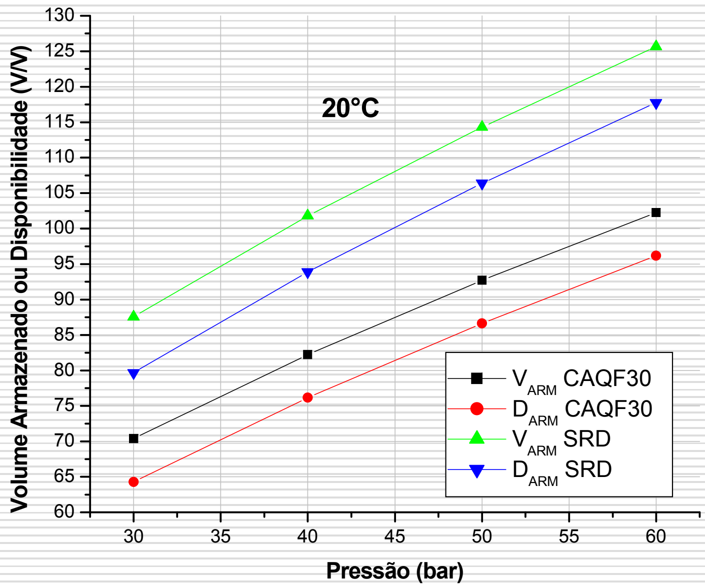
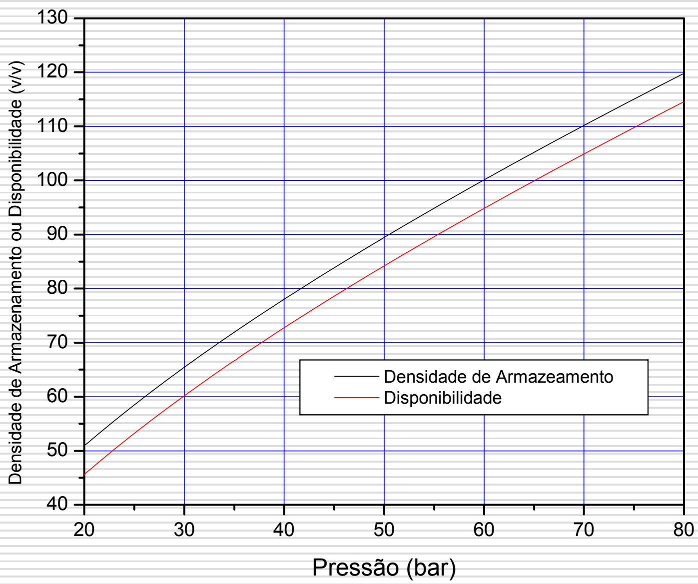
Adsorption capacity and delivery (methane)





$$m_{arm}(p,T) = m_{ex}(p,T) + (V_v) \cdot \rho(p,T)$$

$$V_{arm}(p,T) = \frac{R \cdot T_0}{M \cdot p_0} \cdot m_{arm}(p,T) \cdot \rho_b$$

$$D_{arm} = \frac{R \cdot T_0}{M \cdot p_0} \rho_b \cdot [m_{arm}(p,T) - m_{arm}(p_o,T)]$$

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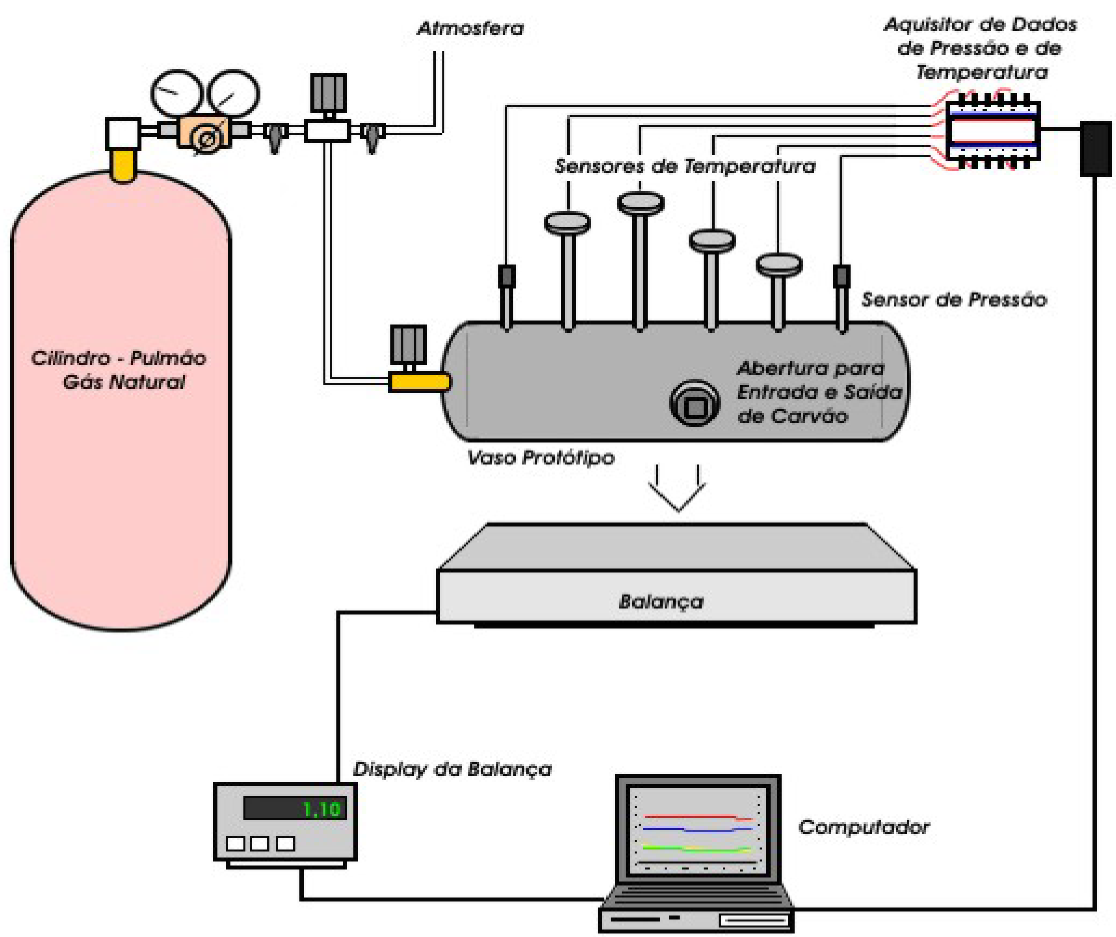
Charge and Discharge Cycles



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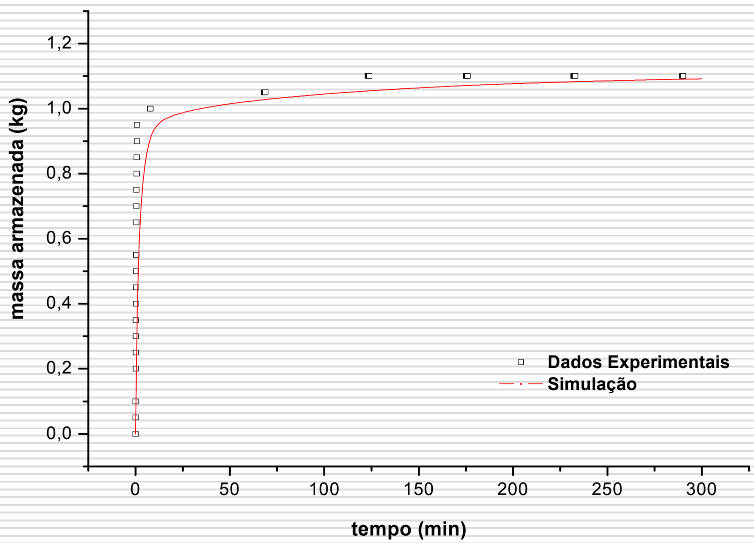
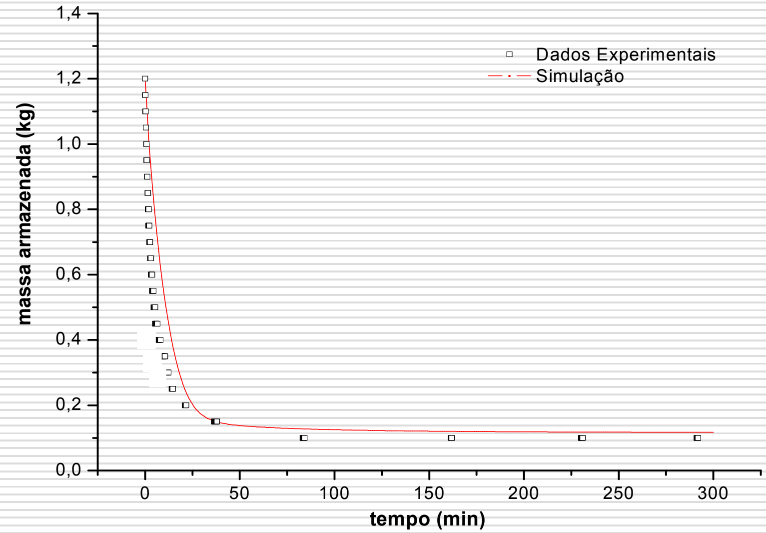
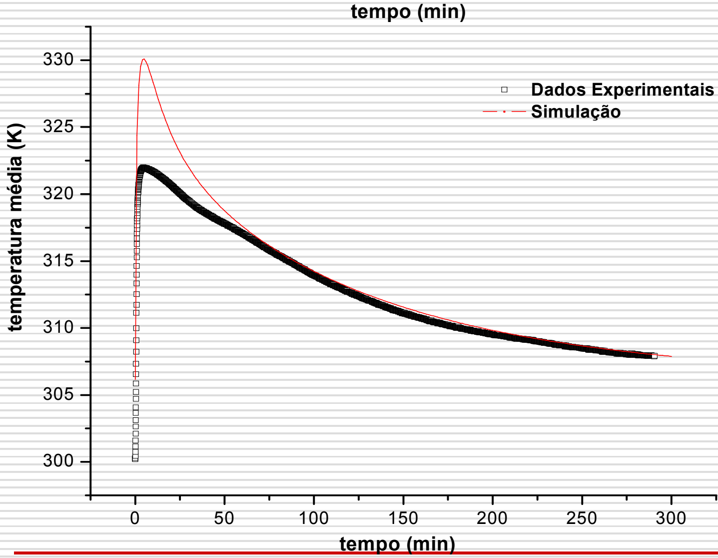
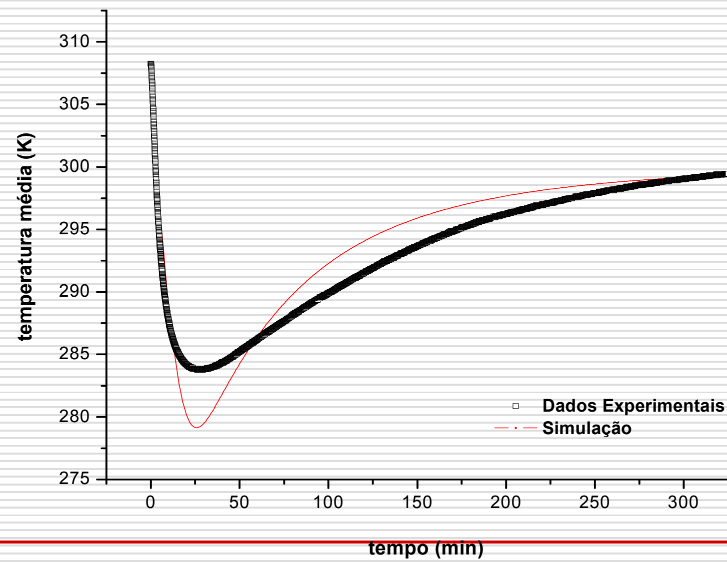
Charge and Discharge Cycles (typical transient behaviour on one cycle)

Bastos-Neto et al., Adsorption 11 (2005) 47









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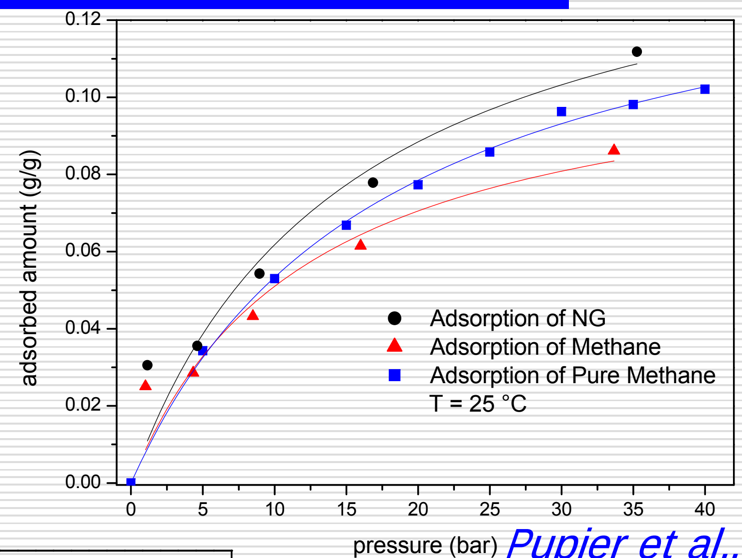
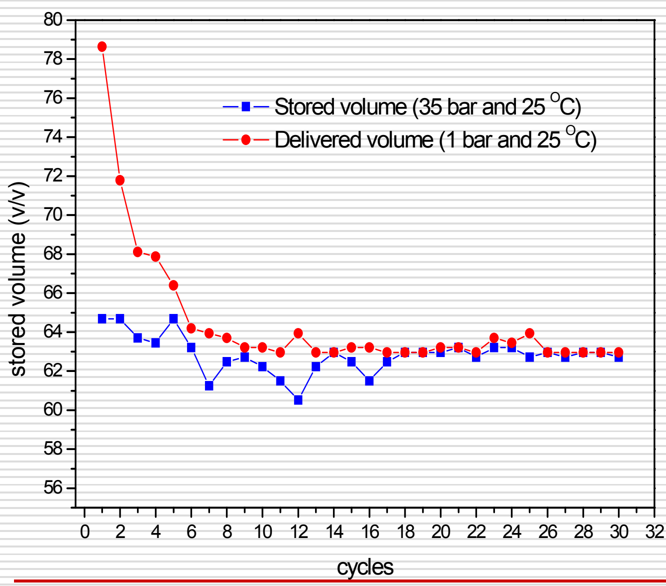
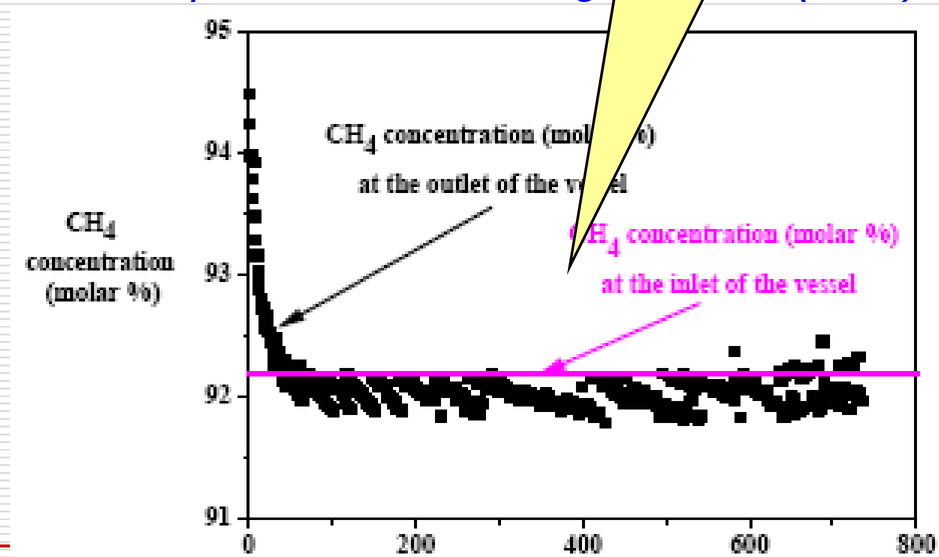
Charge and Discharge Cycles (long-term performance)

Rios et al., unpublished results





Pupier et al., Chem. E... ...) 71



Calorific power of delivered gas varies!!!!

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Charge and Discharge Cycles (long-term performance – fast cycles)

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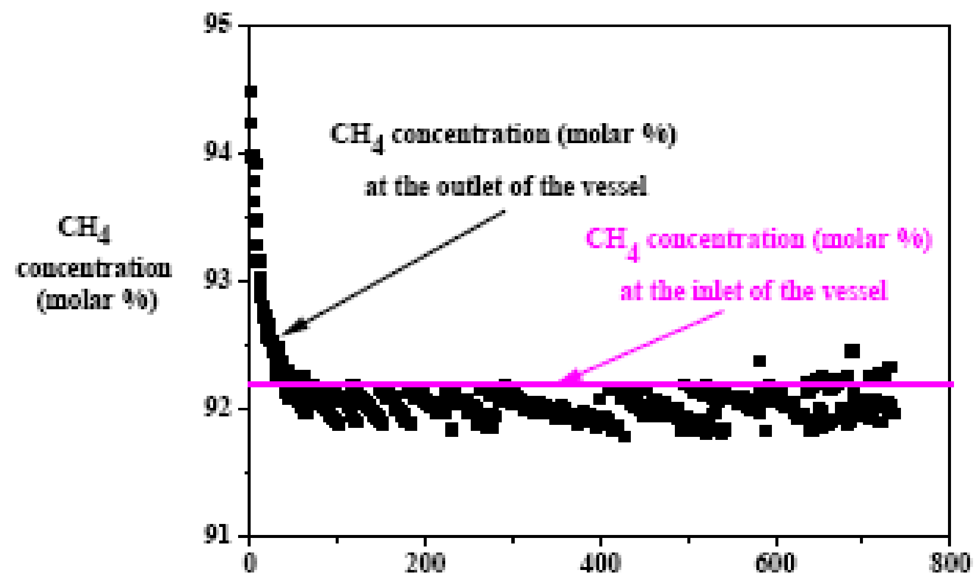
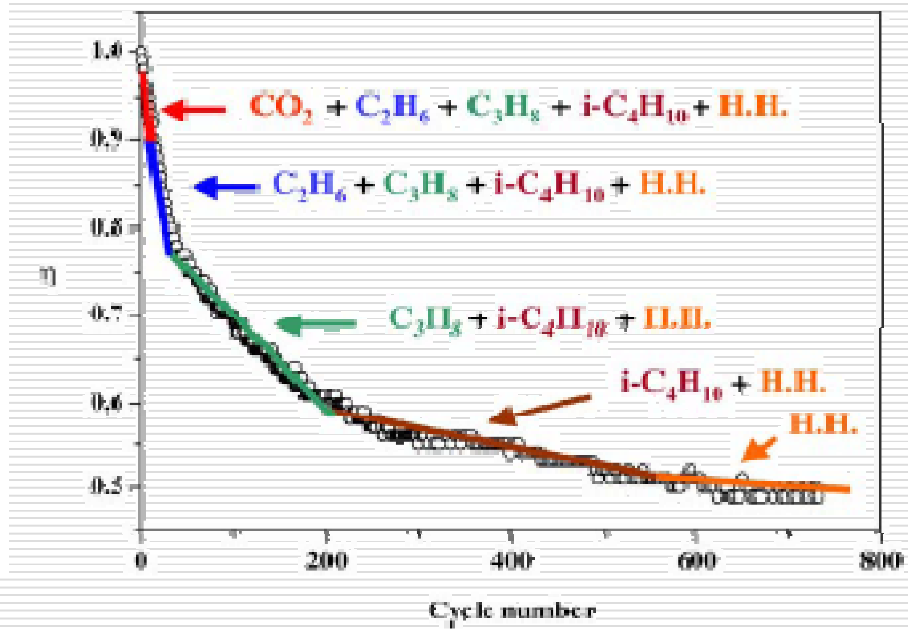
$$\eta = \frac{Q_n}{Q_1}$$





CH_4	N_2	CO_2	C_2H_6	C_3H_8	i-C_4H_{10}	n-C_4H_{10}	i-C_5H_{12}	n-C_5H_{12}	C_{6+}
92.18	1.82	0.75	4.23	0.78	0.13	0.09	0.012	0.006	0.002

Charge and Discharge Cycles (long-term performance) – use of guard beds

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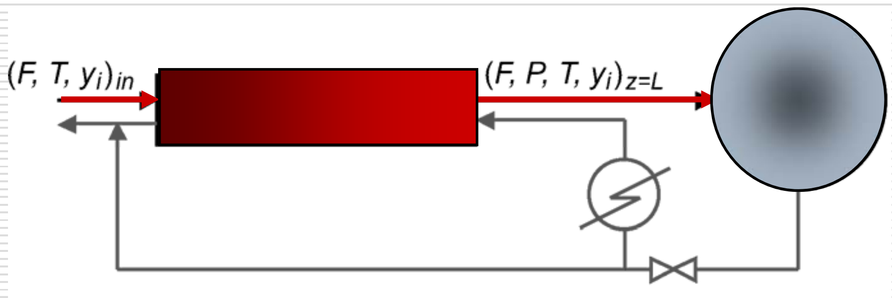
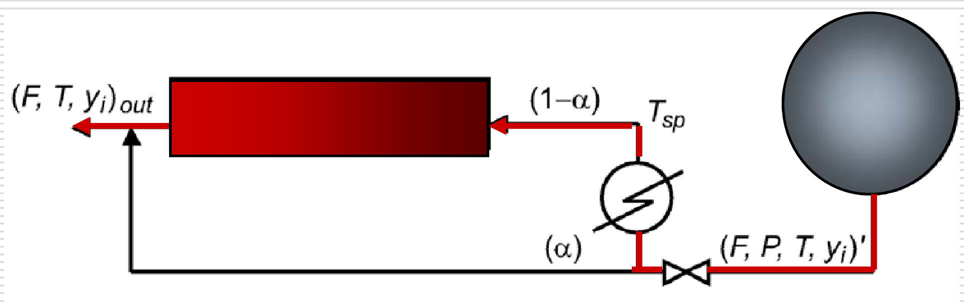
Upon charge of storage vessel, heavy alkanes and odorants are captured in the guard bed



Upon discharge of storage vessel, heavy alkanes and odorants are released from the guard bed



Esteves et al., Adsorption 11 (2005) 905

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Multicomponent adsorption measurements – volumetry w/ GC

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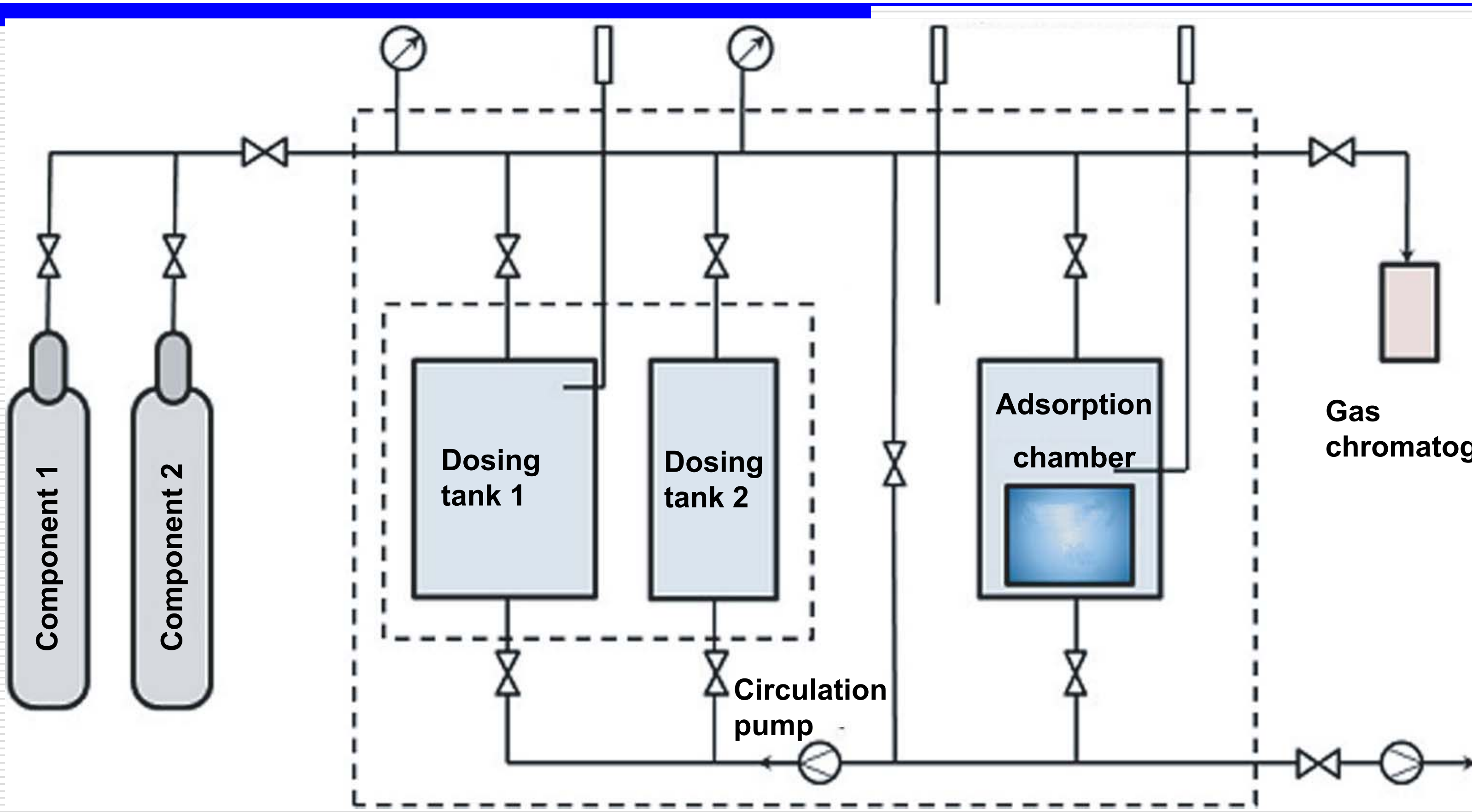

Concentration of fluid phase	Concentration of adsorbed phase	Total amount adsorbed
$\Delta c/c = 1.0$ %	$\Delta c/c = 2 - 5$ %	$\Delta m/m = 1.5$ %

Data for Natural Gas components – monocomponent isotherms

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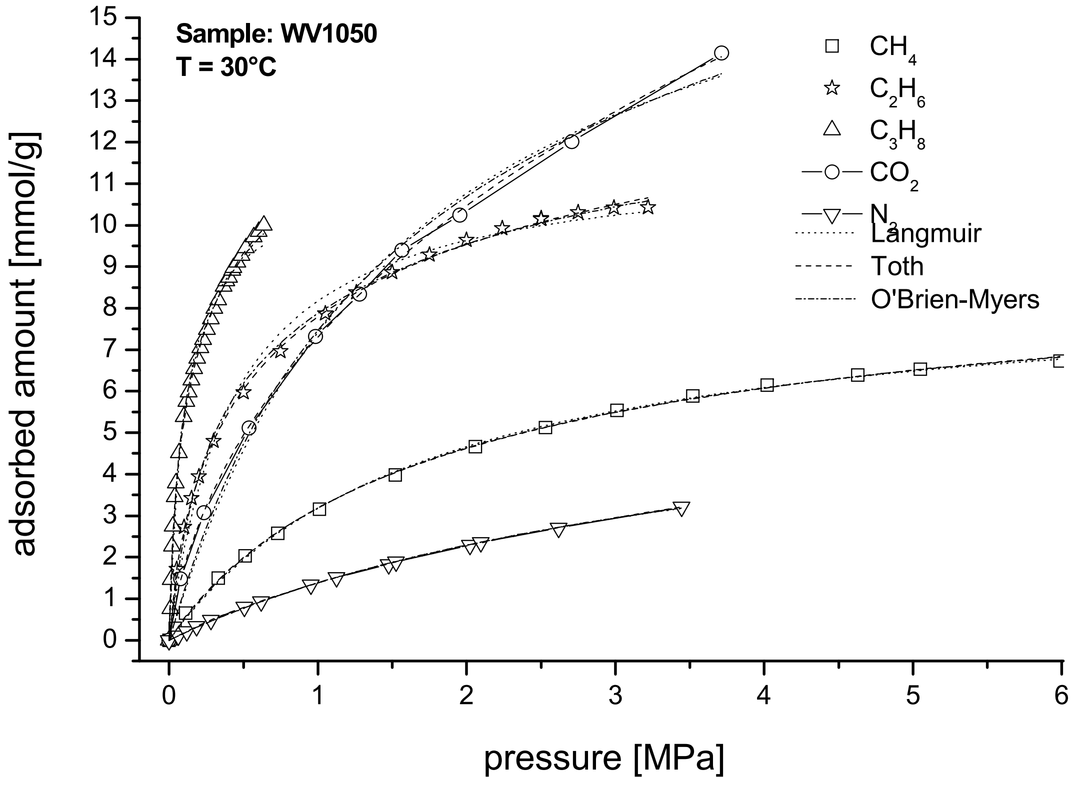

Bazan et al., Adsorption Science and Technology, 2009 (in press)

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Data for Natural Gas components – binary mixtures

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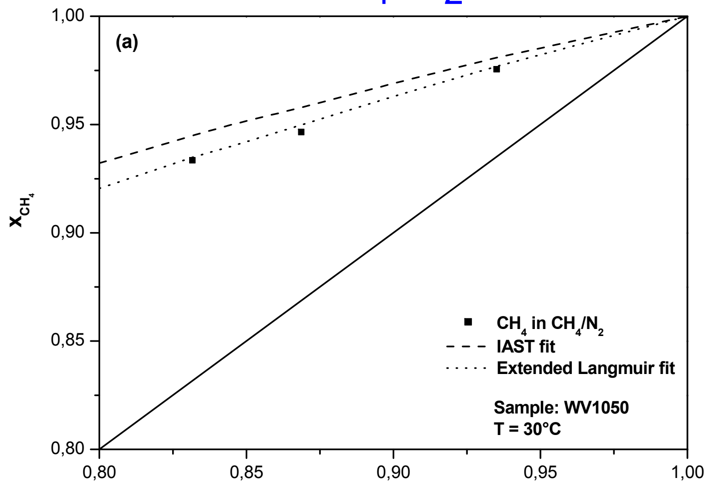
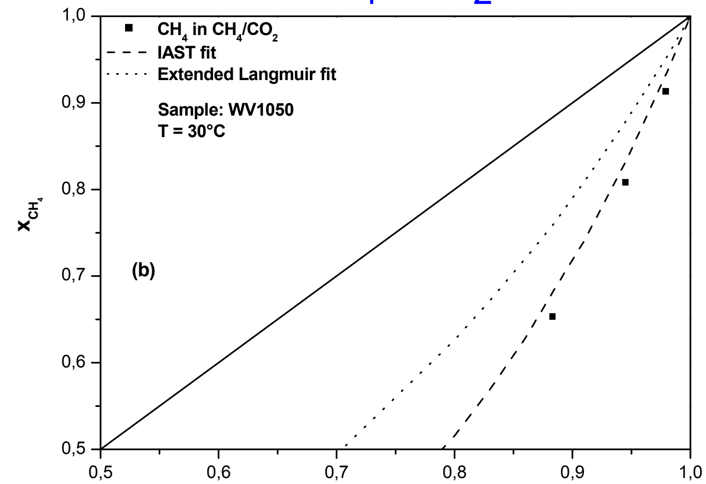
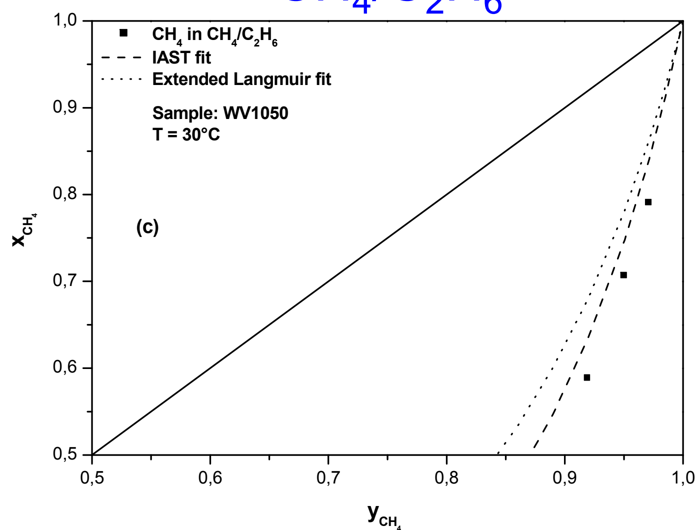
CH_4/N_2



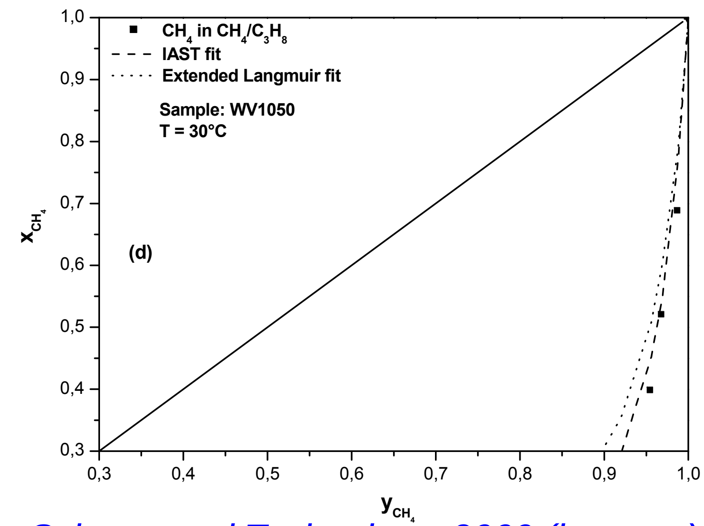
CH_4/CO_2



CH_4/C_2H_6



CH_4/C_3H_8



Bazan et al., Adsorption Science and Technology, 2009 (in press)

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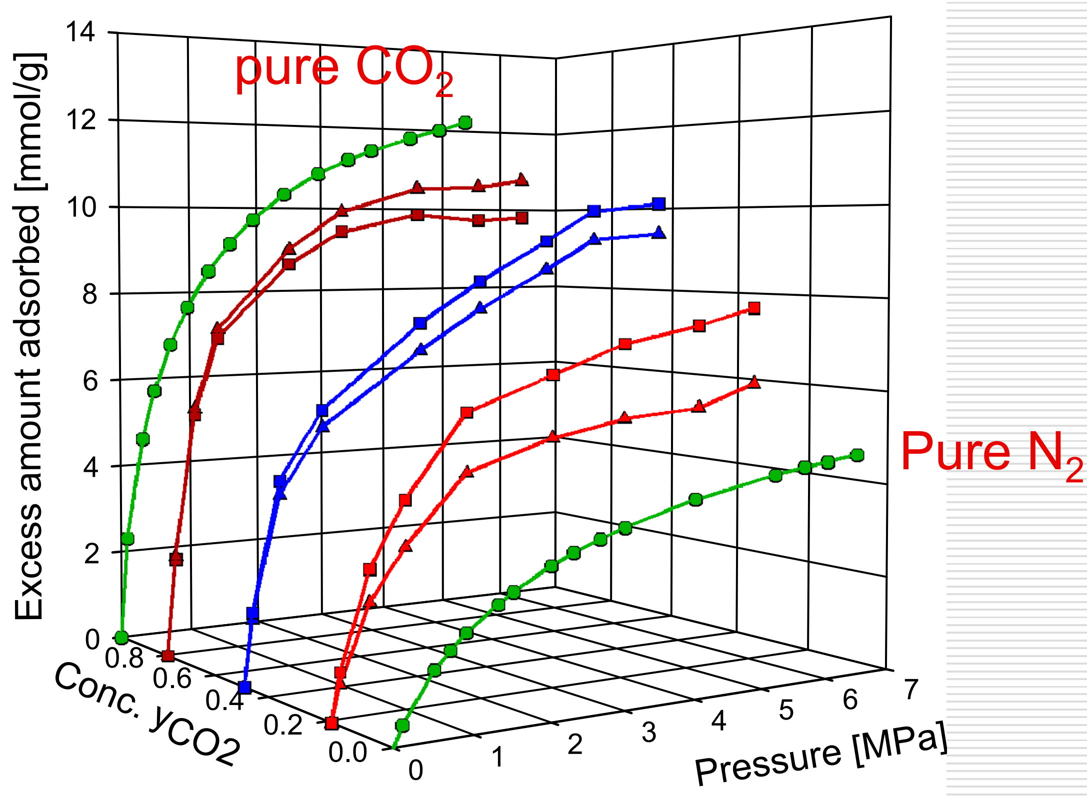
CO2/N2 in AC Norit R1



Bazan and Staudt (unpublished results)

CH4/CO2/N2 in AC Norit R1, T = 298 K

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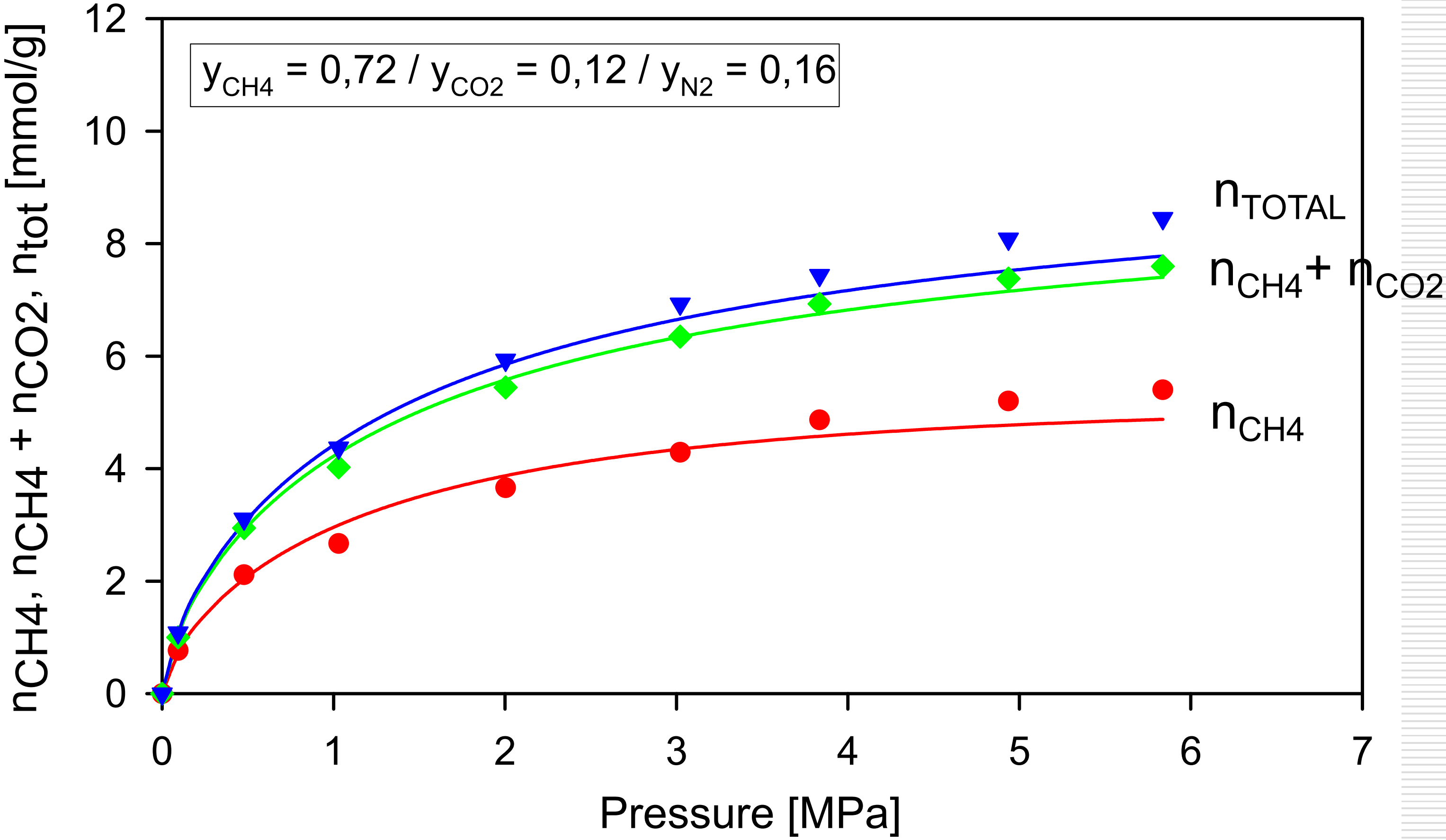

Bazan and Staudt (unpublished results)

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Concluding remarks

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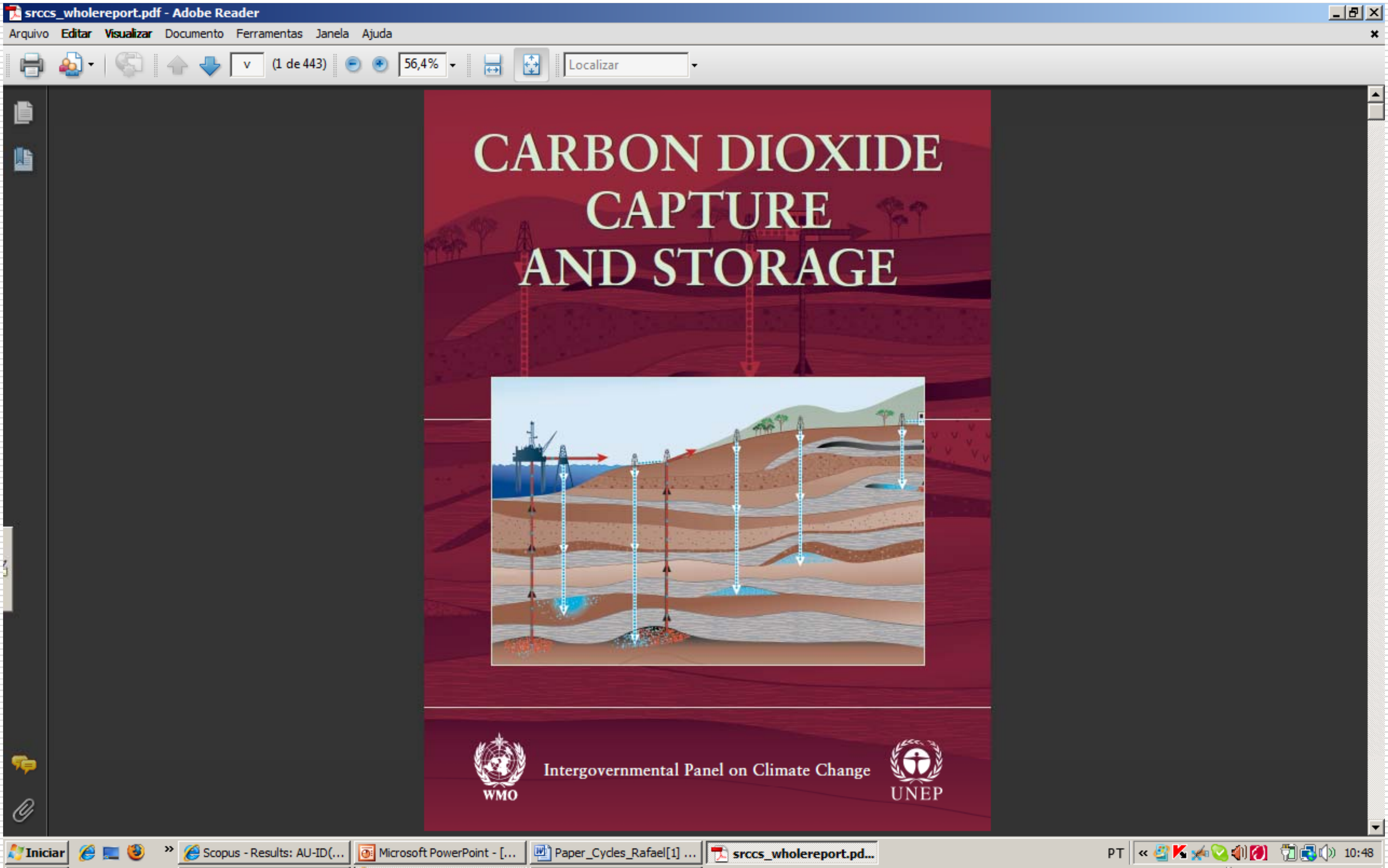
http://www.ipcc.ch



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Some FAQs about CCS

What is CO_2 capture and storage ?

Carbon dioxide (CO_2) capture and storage (CCS) is a process consisting of the separation of CO_2 from industrial and energy-related sources, transport to a storage location and long-term isolation from the atmosphere.

What are the characteristics of CCS?

Capture of CO_2 can be applied to large point sources. The CO_2 would then be compressed and transported for storage in geological formations, in the ocean, in mineral carbonates, or for use in industrial processes.

In most CCS systems, the cost of capture (including compression) is the largest cost component.

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Possible CCS scenarios

IPCC Special Report on CCS, Cambridge Univ. Press, 2005

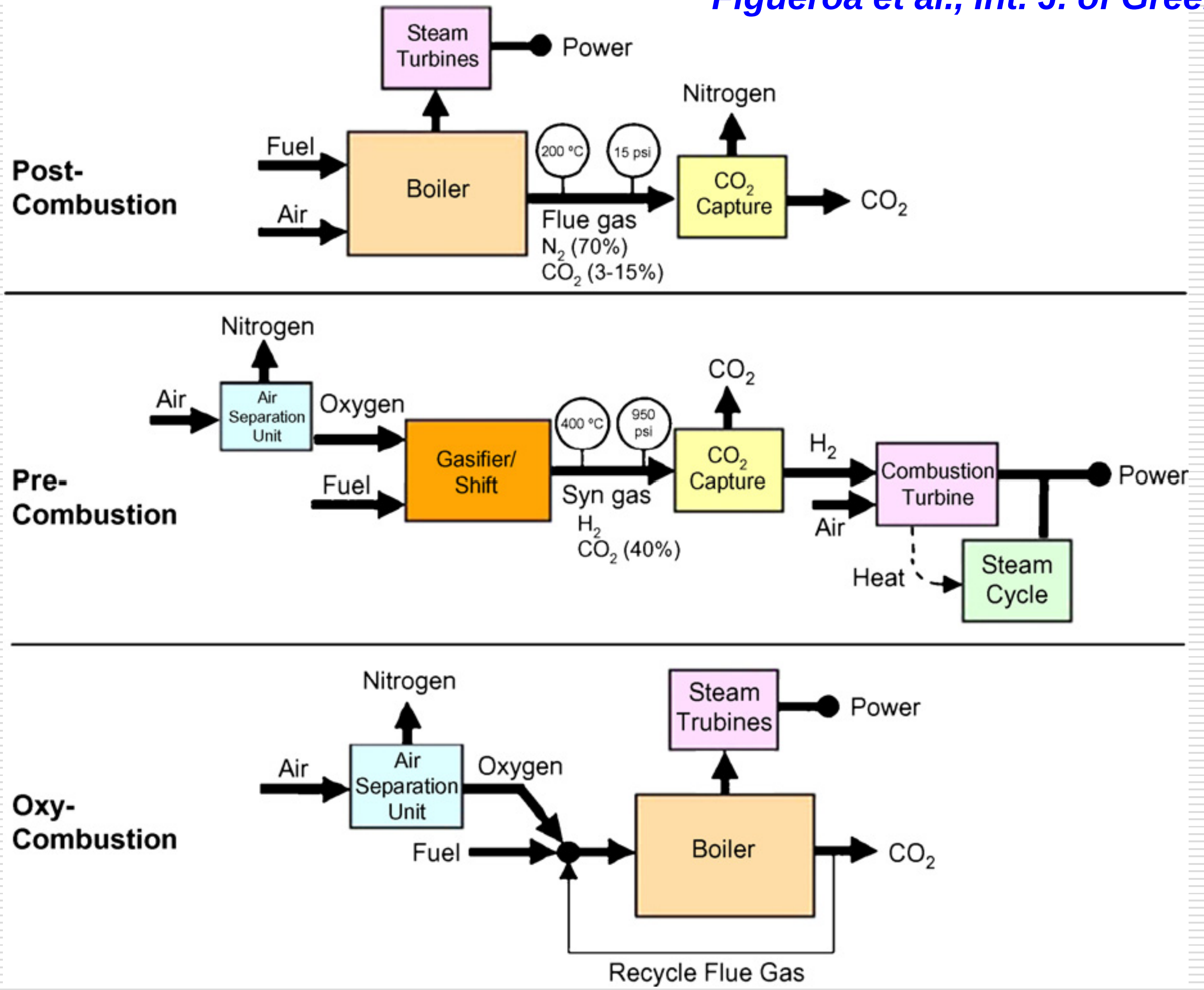
Table TS.9. 2002 Cost ranges for the components of a CCS system as applied to a given type of power plant or industrial source. The costs of the separate components cannot simply be summed to calculate the costs of the whole CCS system in US$/$CO_2$ avoided. All numbers are representative of the costs for large-scale, new installations, with natural gas prices assumed to be 2.8-4.4 US$ GJ^{-1} and coal prices 1-1.5 US$ GJ^{-1}.

CCS system components	Cost range	Remarks
Capture from a coal- or gas-fired power plant	15-75 US$/t$CO_2$ net captured	Net costs of captured CO_2, compared to the same plant without capture.
Capture from hydrogen and ammonia production or gas processing	5-55 US$/t$CO_2$ net captured	Applies to high-purity sources requiring simple drying and compression.
Capture from other industrial sources	25-115 US$/t$CO_2$ net captured	Range reflects use of a number of different technologies and fuels.
Transportation	1-8 US$/t$CO_2$ transported	Per 250 km pipeline or shipping for mass flow rates of 5 (high end) to 40 (low end) MtCO_2 yr^{-1}.
Geological storage[a]	0.5-8 US$/t$CO_2$ net injected	Excluding potential revenues from EOR or ECBM.
Geological storage: monitoring and verification	0.1-0.3 US$/t$CO_2$ injected	This covers pre-injection, injection, and post-injection monitoring, and depends on the regulatory requirements.
Ocean storage	5-30 US$/t$CO_2$ net injected	Including offshore transportation of 100-500 km, excluding monitoring and verification.
Mineral carbonation	50-100 US$/t$CO_2$ net mineralized	Range for the best case studied. Includes additional energy use for carbonation.

[a] Over the long term, there may be additional costs for remediation and liabilities.

CO_2 capture systems

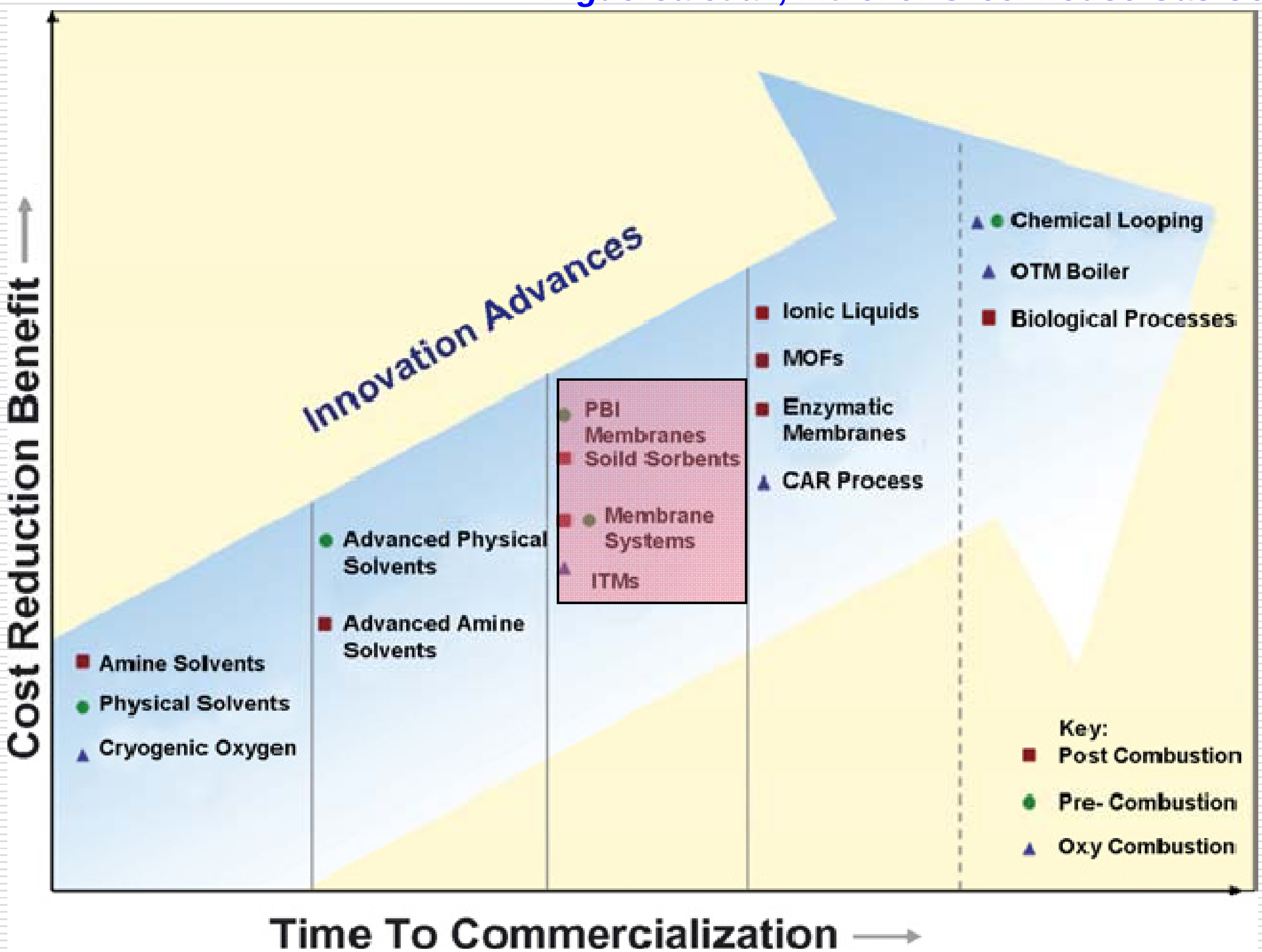
Figueroa et al., Int. J. of Greenhouse Gas Contr. 2 (2008) 9



The concentration of CO_2 in the gas stream, the pressure of the gas stream and the fuel type (solid or gas) are important factors in selecting the capture system.

Cost reduction benefits Vs. Time to commercialization

Figueroa et al., Int. J. of Greenhouse Gas Contr. 2 (2008) 9



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Capture Toolbox

IPCC Special Report on CCS, Cambridge Univ. Press, 2005

Separation task	Process Streams		Post-combustion Capture		Oxy-fuel Combustion		Pre-combustion Capture	
	CO_2 / CH_4		**CO_2 / N_2**		**O_2 / N_2**		**CO_2 / H_2**	
Capture technol.	Current	Emerging	Current	Emerging	Current	Emerging	Current	Emerging
Absortion	Physical Solvs. Chemical Solvs.	Improved solvs. And proc design Contact equipm	Chemical Solvents	Improved solvs. And proc design Contact equipm	n.a.	Biomimetic solvents i.e. Hemo globyn derivatives		Improved solvs. And proc design Contact equipm
Membranes	Polymeric	Ceramic Enhanced transport Carbon	Polymeric	Ceramic Enhanced transport Carbon	Polymeric	Ion-transport membranes	Polymeric	Ceramic Palladium Reactors Contactors
Adsorption	**Zeolites Activ. Carbon**		**Zeolites Activ. Carbon**	**Carbonate Carbon-based sorbents**	**Zeolites Activ. Carbon**	**Adsorbents for O2/N2 separation perovskite**	**Zeolites Activ. Carbon Alumina**	**Carbonates Hydrotalcites Silicates MOFs**
Cryogenic	Ryan-Holmes Process		Liquefaction	Hybrid processes	Distillation	Improved destillation	Liquefaction	Hybrid processes

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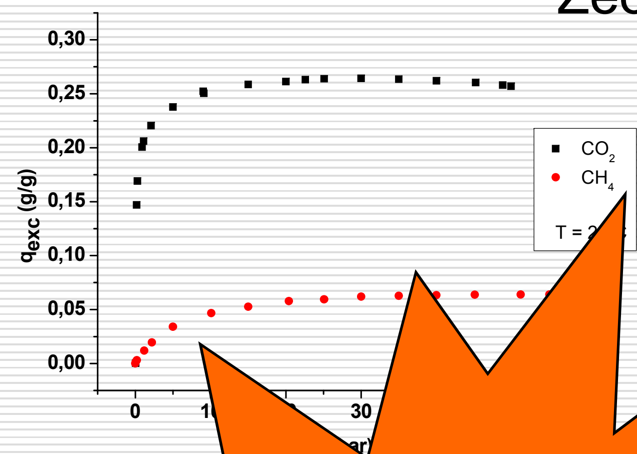
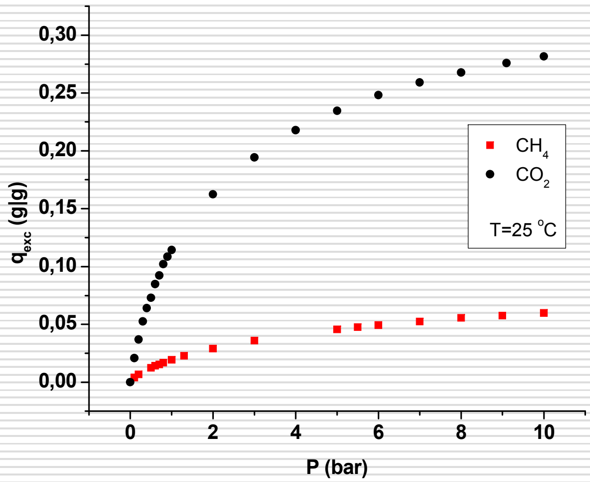
Classical sorbents for CO_2 adsorption

(Bezerra et al., 2009, unpublished results)

Zeolite 13 X

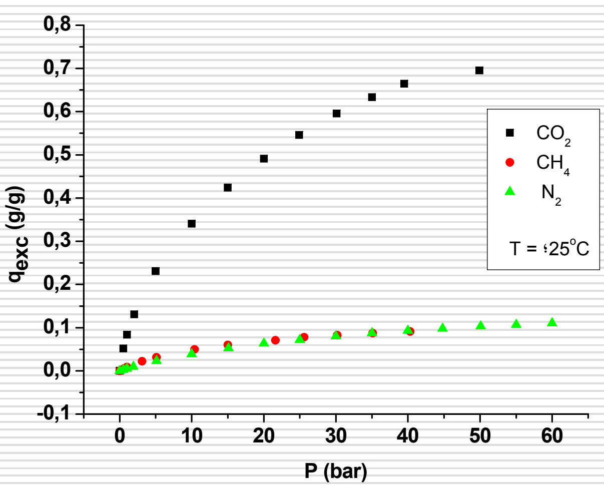


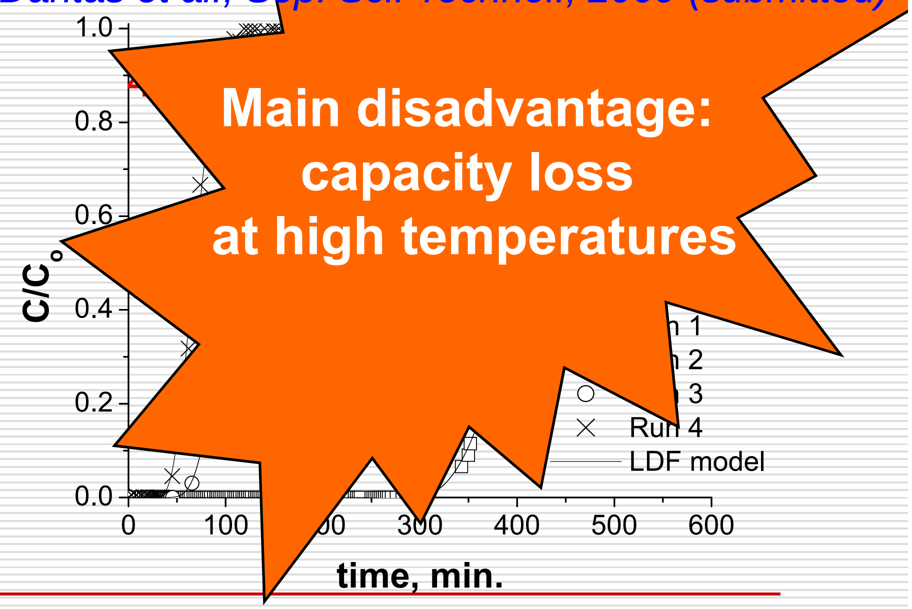
AC Norit CN2



Dantas et al., Sep. Sci. Technol., 2009 (submitted)

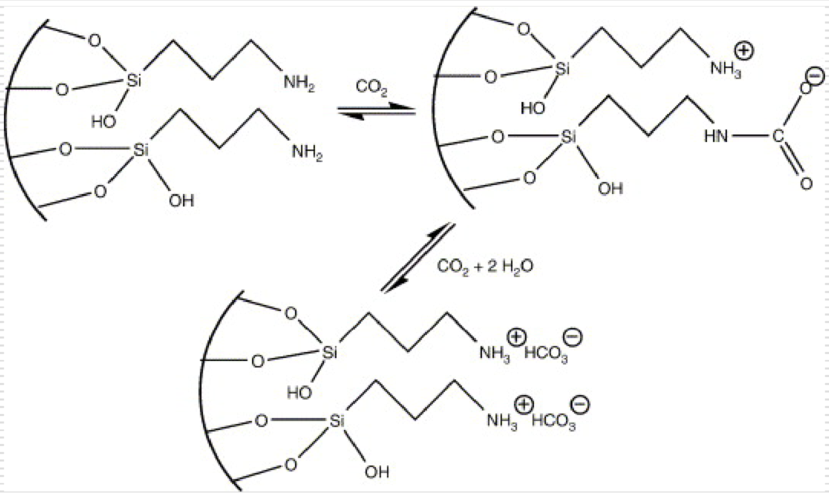
AC Westvaco 1050



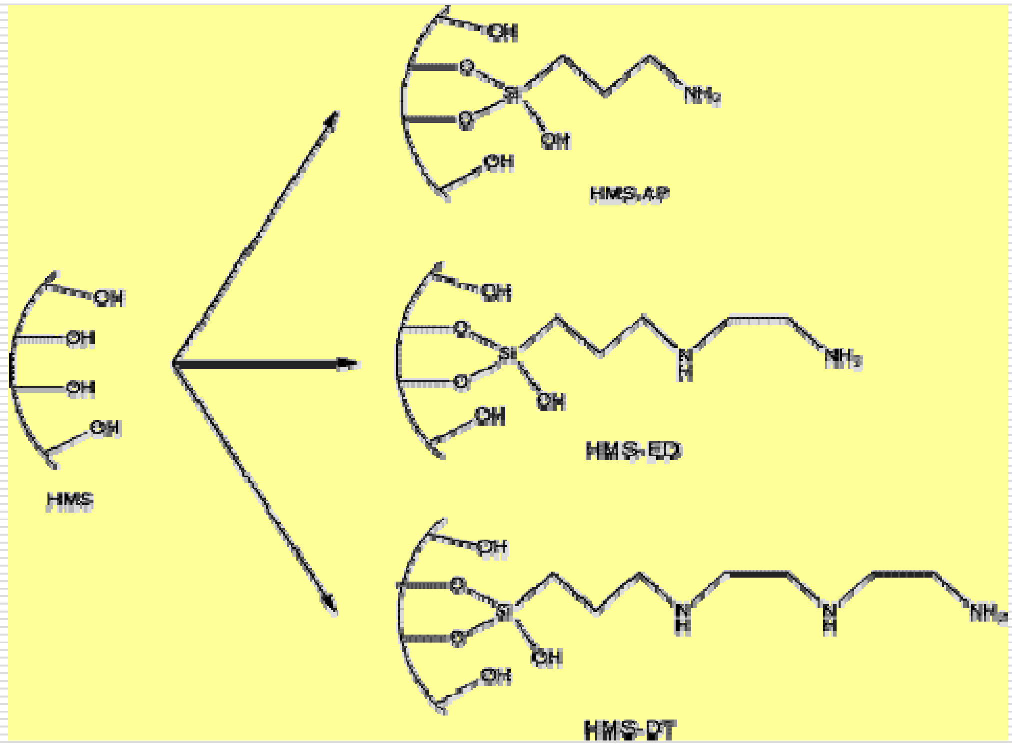


Emerging sorbents

- Amines incorporated on inorganic supports (silicas, zeolites)



Knowles et al., Fuel Proc. Technol. 86 (2005) 1435



Knowles et al., Ind. Eng. Chem. Res. 45 (2006) 2626

Emerging sorbents

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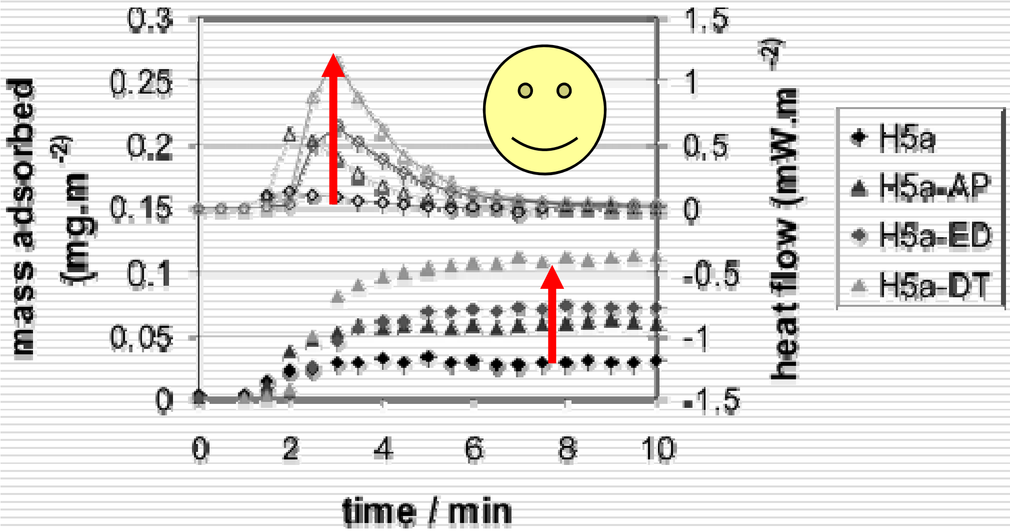
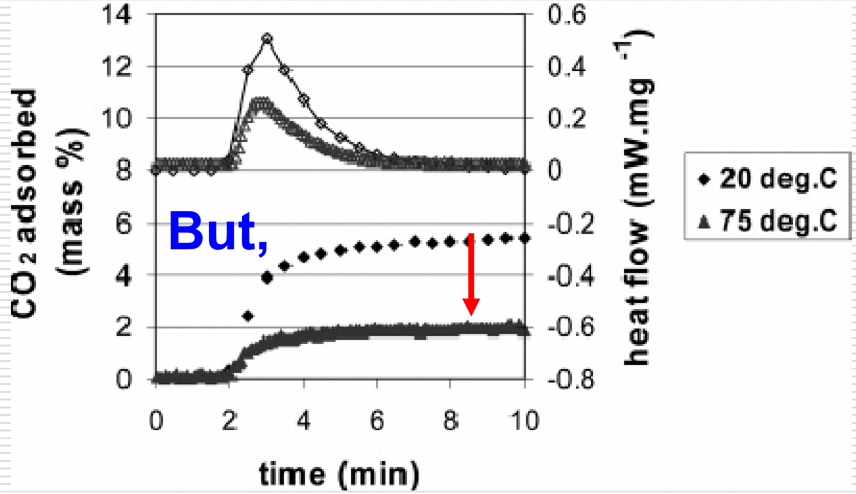
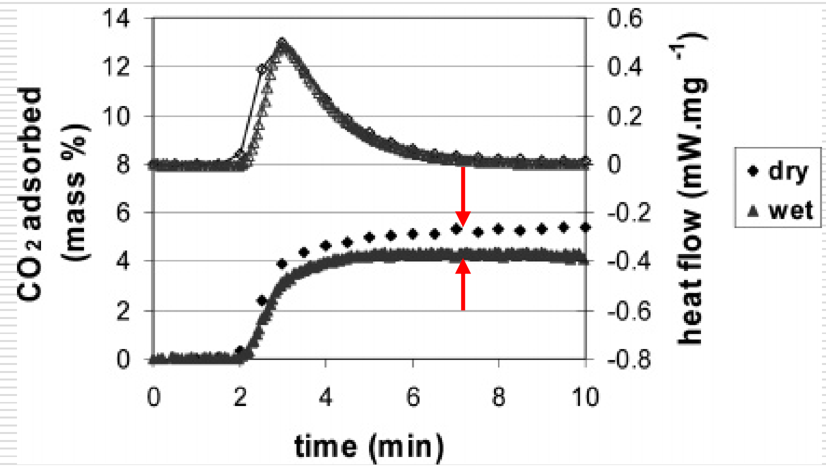




AP – aminopropylsilyl
ED - ethylenediamine[propyl(silyl)]-
DT - diethylenetriamine[propyl(silyl)]-



Knowles et al., Ind. Eng. Chem. Res. 45 (2006) 2626

Emerging sorbents

Chatti et al., Microp and Mesop Materials, 2009 (in press)

Table 6: Equilibrium adsorption capacities of zeolite 13X and MEA modified zeolite 13X (50 w%) at 75ºC and 1 bar gauge pressure.

	Equilibrium adsorption capacity (mg/g)
13X matrix	37.33
13X/MEA	48.64

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Emerging sorbents

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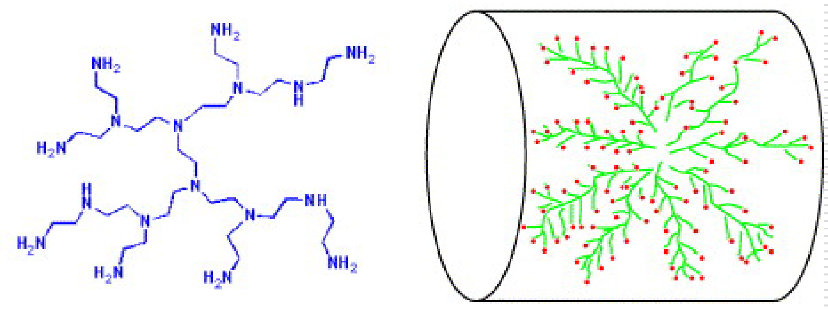
Song, Catalysis Today 115 (2006) 2

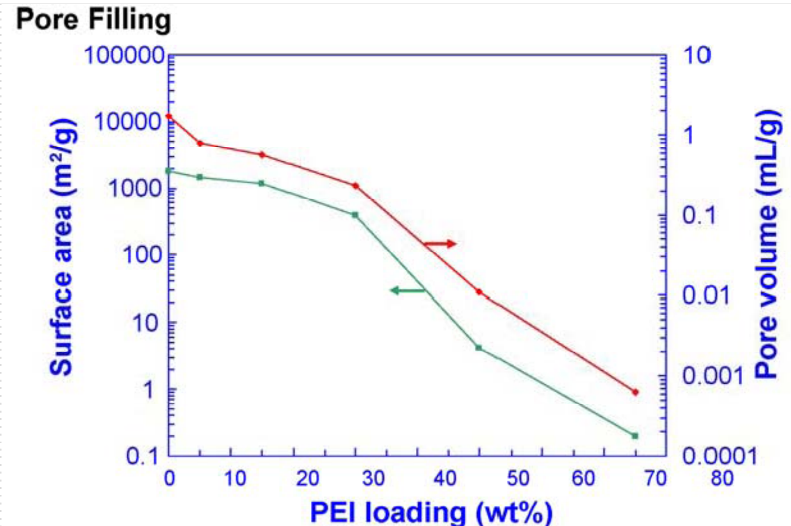
"Molecular basket" adsorbents

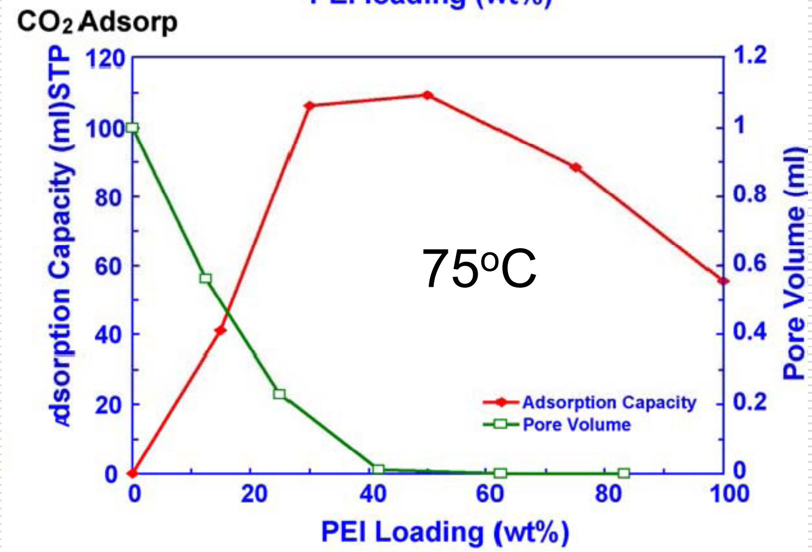
Fill pores of mesoporous matrices with PEI, which would capture CO_2 in terminal amino- groups



Polyethylenimine (PEI)   PEI in Nano-porous Channel

- Large pore volume nanoporous support can store large amount of CO_2
- Branched CO_2-affinity polymers provide adsorption sites
- Branched amine facilitate the desorption
- Synergic effect on the CO_2 adsorption capacity and adsorption kinetic between nanoporous support and polyethylenimine





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Emerging sorbents

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Table 2
Comparison of CO_2 adsorption performance of "molecular basket" adsorbent and other adsorbents

Adsorbents	Temp (°C)	Pressure (atm)	Adsorption capacity (mg CO_2/g adsorbent)	CO_2/N_2 or CO_2/CH_4 selectivity	Ref.
Si-MCM-41	25	1	27.3	–	This study
Si-MCM-41	75	1	8.6	–	This study
Si-MCM-41	75	0.149	6.3	2.9 (CO_2/N_2)	[51]
Al-MCM-41-100	75	1	7.6	–	This study
Al-MCM-41-500	75	1	7.5	–	This study
Si-MCM-41-PEI-50	25	1	32.9	–	This study
Si-MCM-41-PEI-50	75	1	112	–	This study
Si-MCM-41-PEI-50	75	0.149	89.2	>1000 (CO_2/N_2)	[51]
Al-MCM-41-100-PEI-50	75	1	127	–	This study
Al-MCM-41-500-PEI-50	75	1	121	–	This study
Zeolite 13 X	25	1	168	–	[13]
Zeolite 4A	25	1	135	–	[13]
Activated carbon	25	1	110	–	[13]
Norit RBI activated carbon	21.5	1	108	~2 (CO_2/CH_4)	[28]
Norit RBI activated carbon	75	1	40	~2 (CO_2/CH_4)	[28]
Activated carbon	20	1	88	~ 2 (CO_2/CH_4)	[24]
Norit RBI activated carbon	25	1	140.8	~1.9 (CO_2/CH_4)	[29]
PEI-silica gel	75	1	78.1	–	This study
PEI-polymer	~50	0.02	~40	–	[44]

Xu et al. Microp Mesop Mat 62 (2003) 29

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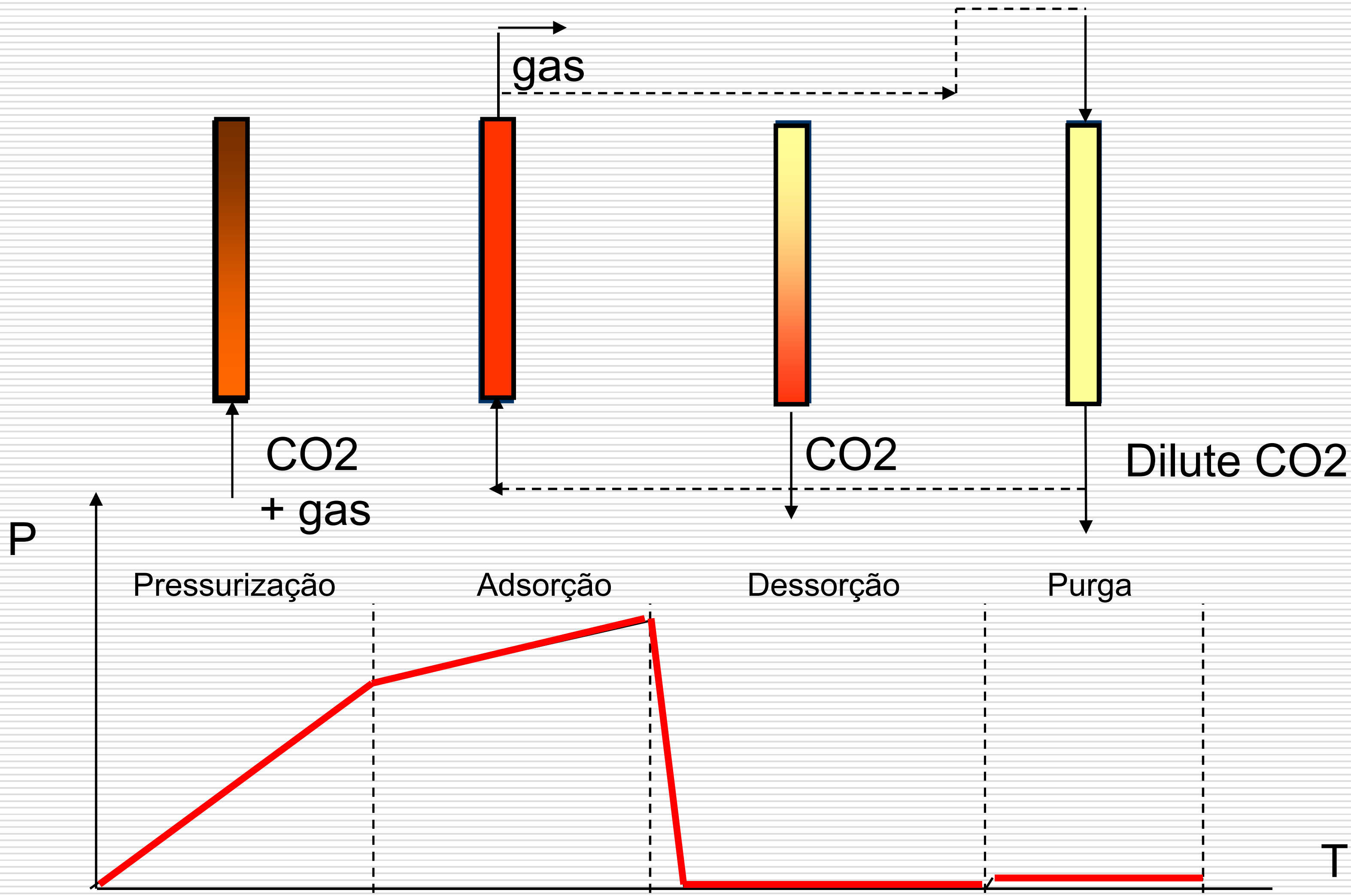
Steps of a one-column PSA cycle



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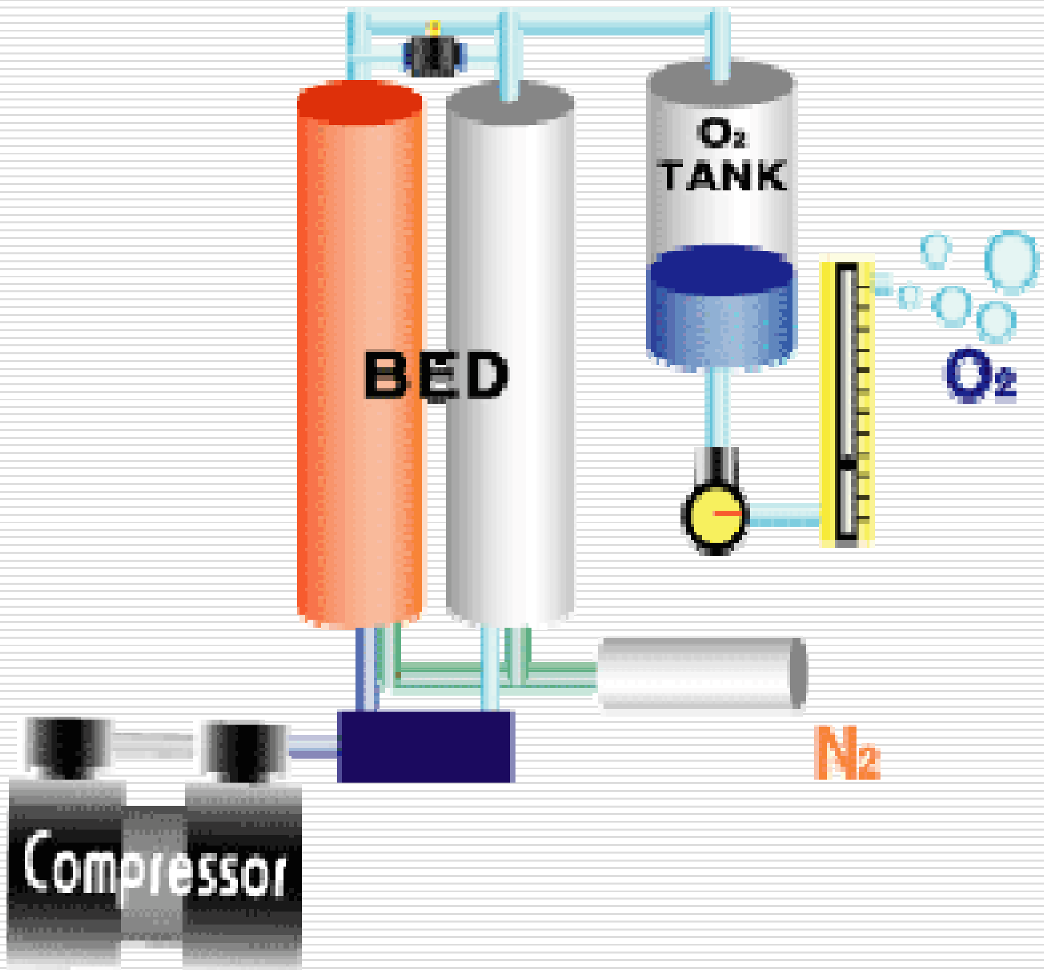
A two-column PSA for air separation



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Basic Principles of a PSA process – column dynamics

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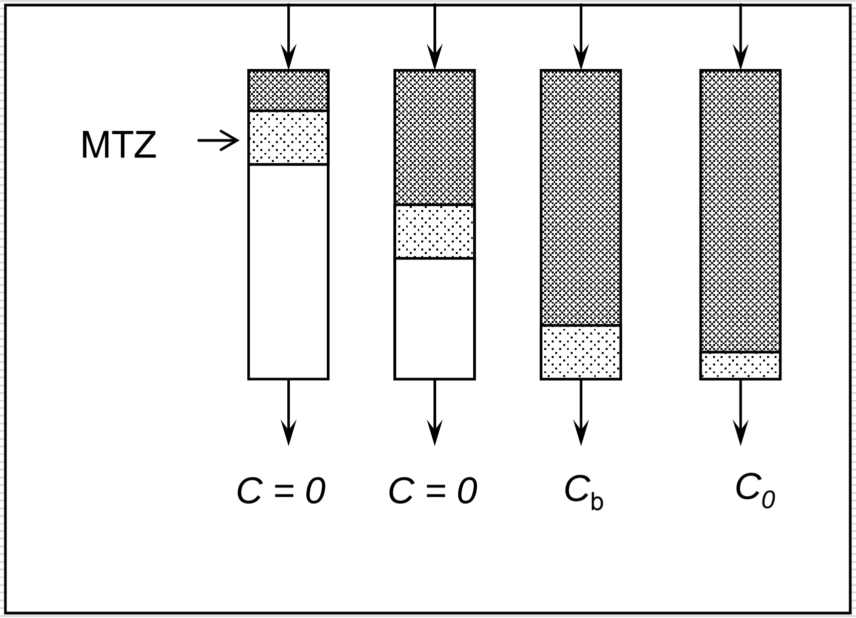
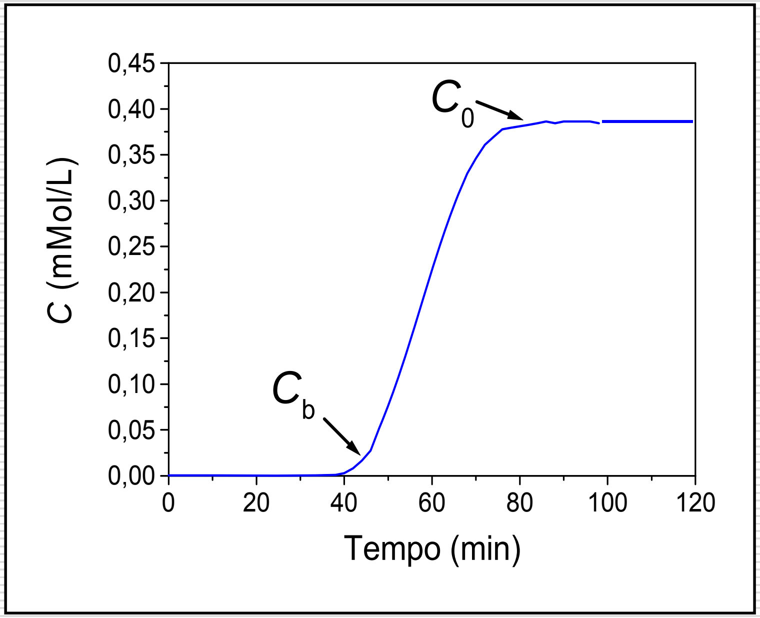
Fixed bed – unit cell of Pressure Swing Adsorption (PSA) processes

Adsorption front progression along the bed



Breakthrough curve
Concentration history in column outlet



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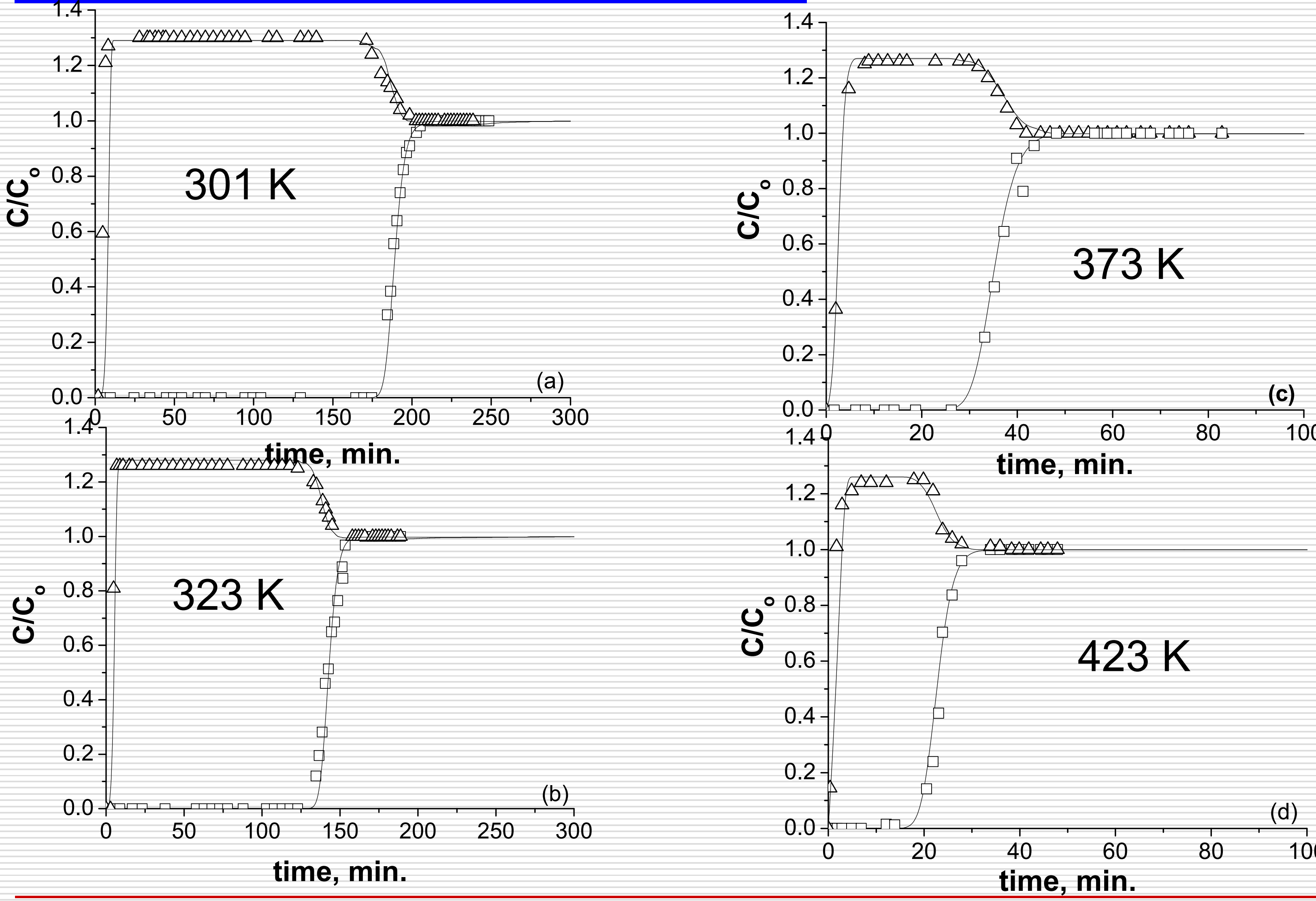
CO_2(□) / N_2(△) breakthrough curves

Dantas et al., Sep. Sci. Technol., 2009 (submitted)



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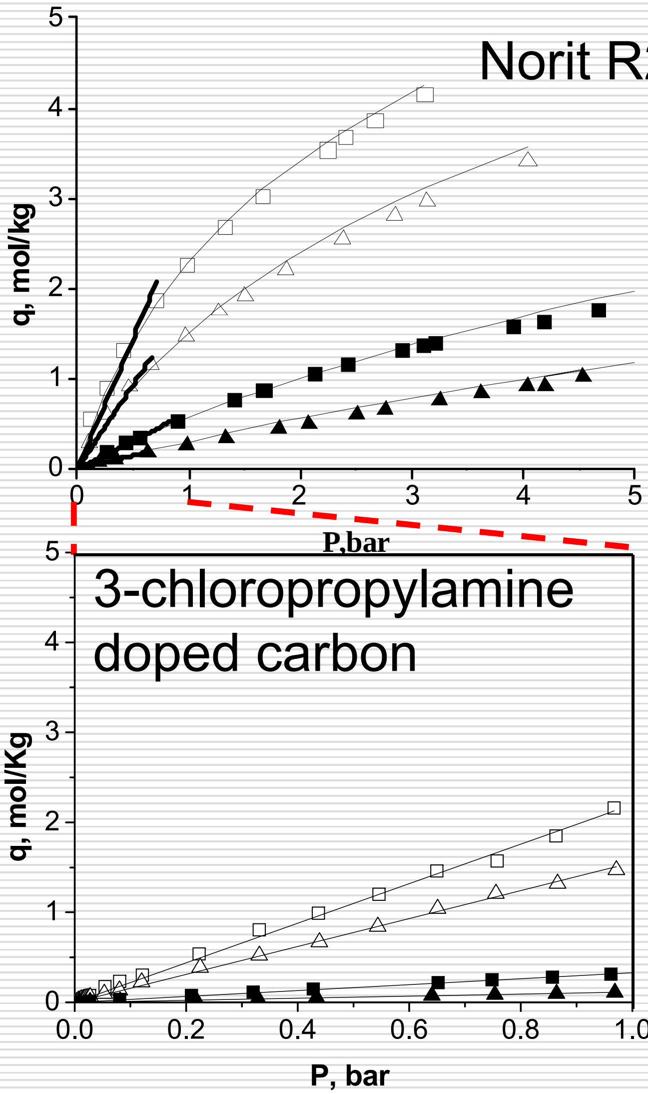
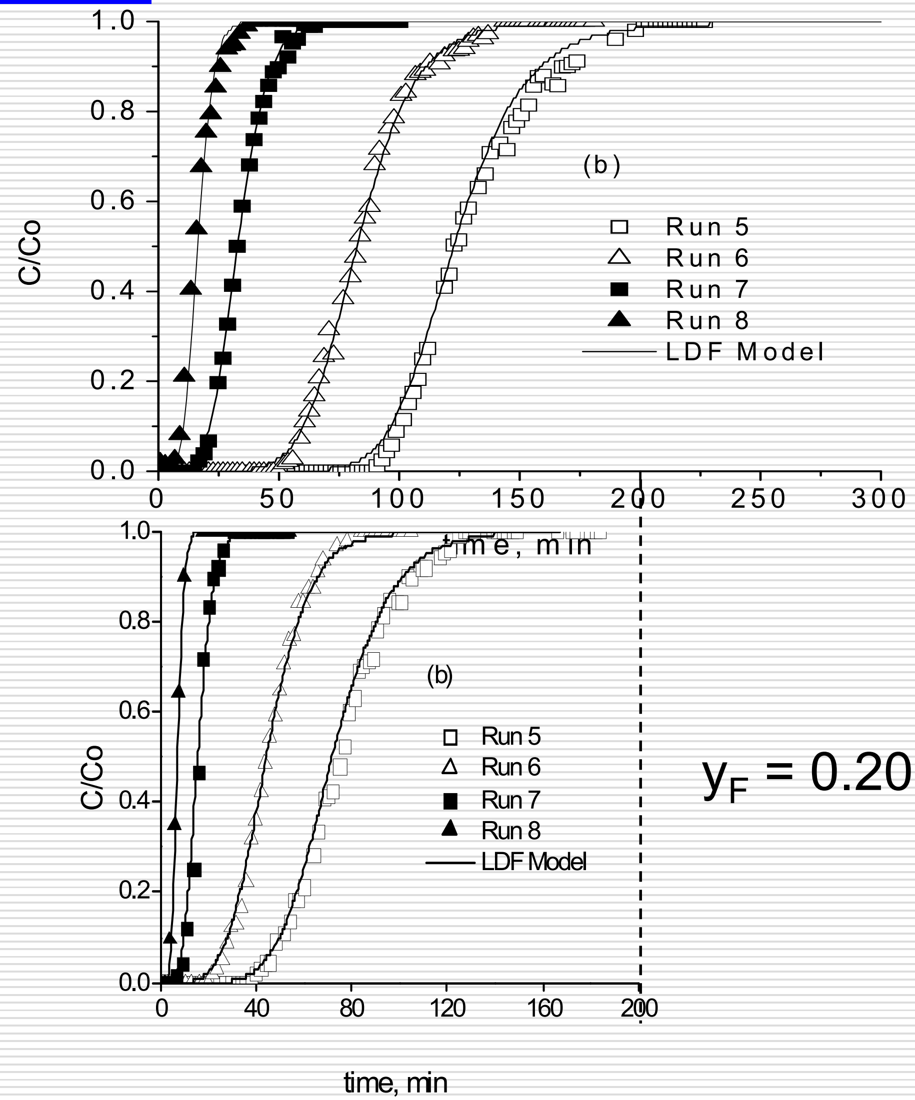
Adding amines to activated carbons

Dantas et al., Sep. Sci. Technol., 2009 (submitted)





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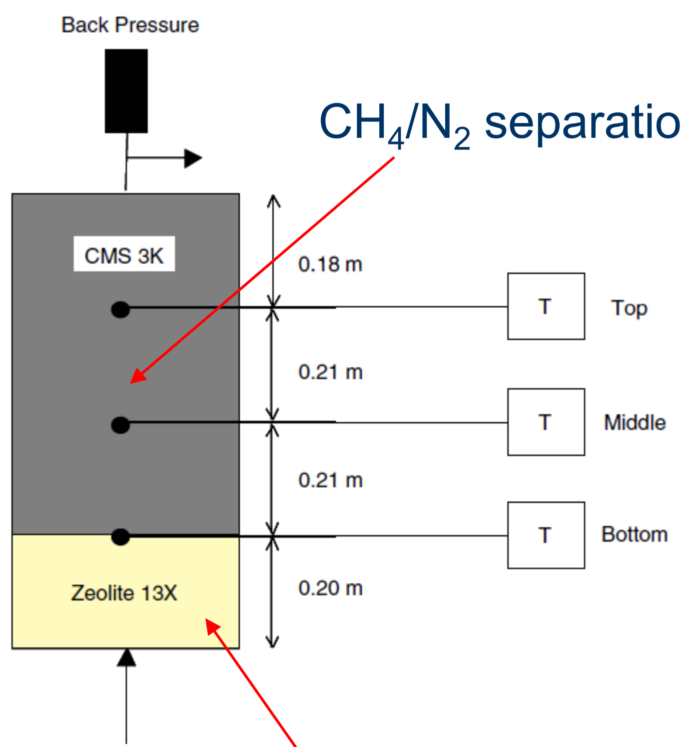
Improvements in process design

DEQ-UFC 15 Anos

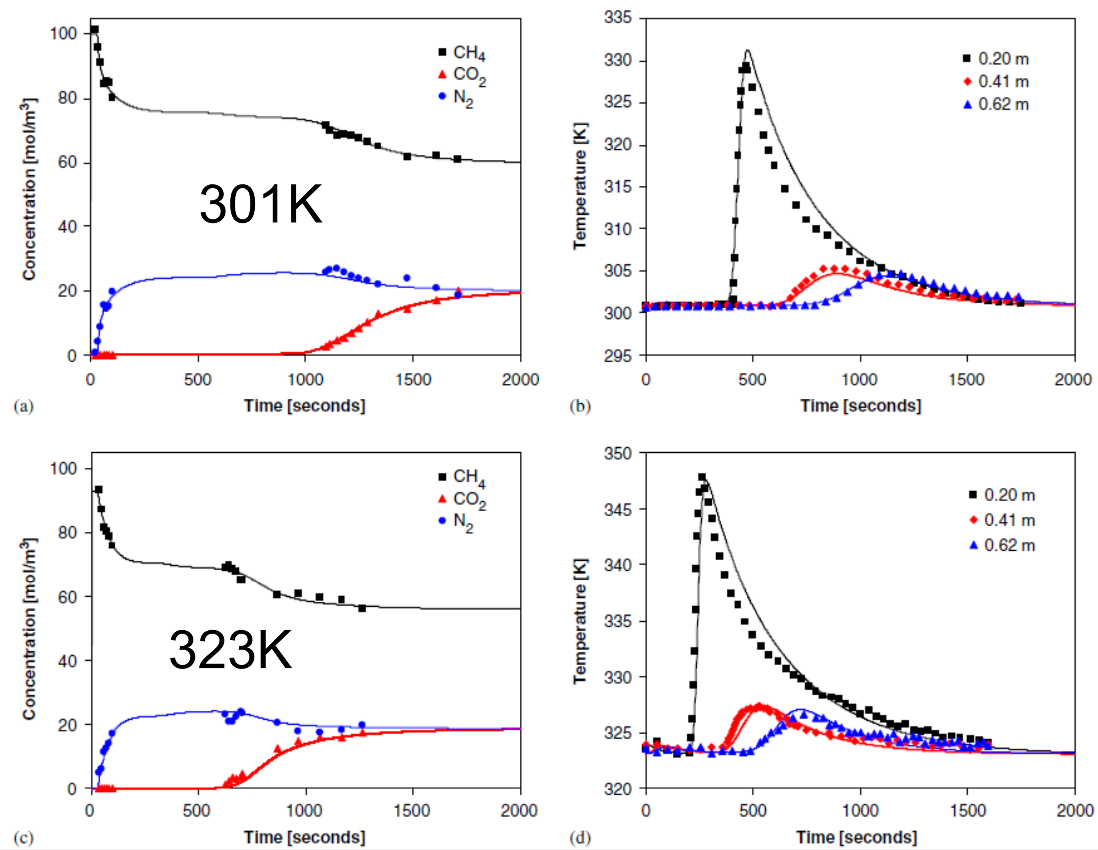
S. Cavenati et al., Chem. Eng. Sci. 61 (2006) 3893

Layered beds

Natural gas purification



CH_4/N_2 separation



CO_2 adsorption

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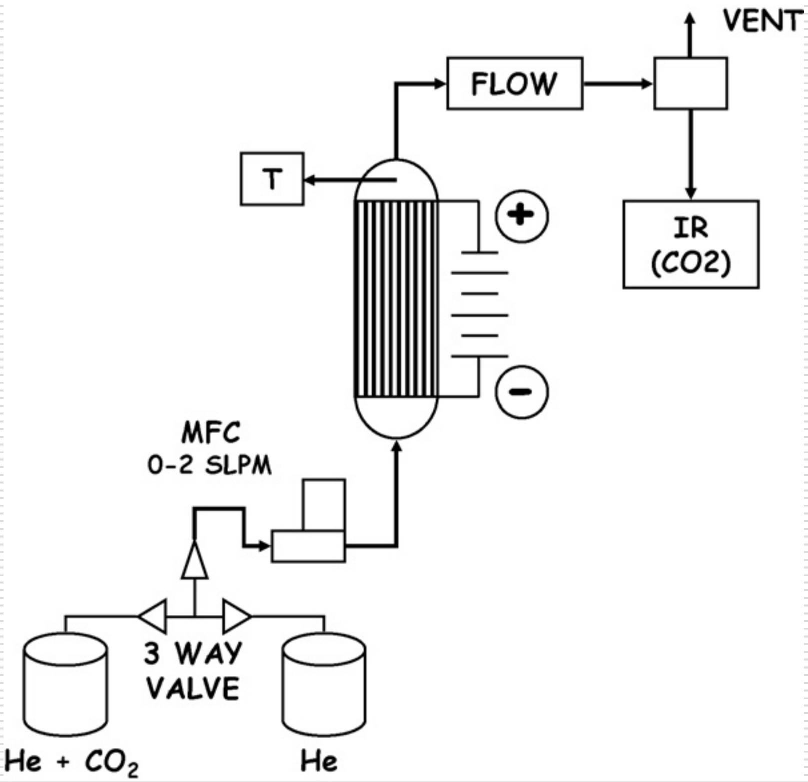
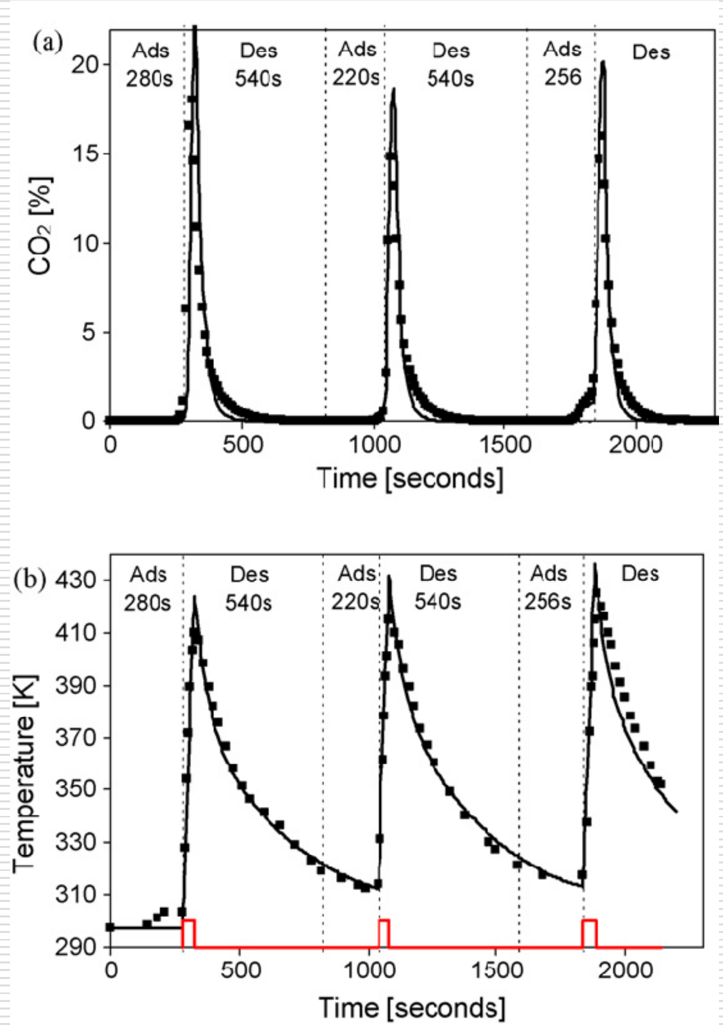
Improvements in process design – Electric Swing Adsorption





CO_2 concentration (4X achieved)

Activated carbon honey comb

Grande and Rodrigues, Int. J. of Greenhouse Gas Cont. 2 (2008) 194

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Concluding remarks

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Design, optimization and control of storage and capture adsorption-based systems relies on:

- Materials development (field for activated carbon and eventually mesoporous matrices)
- Accurate adsorption measurements (single and multicomponent)
- Innovative process schemes
- Modeling (simplicity X accuracy) tools, from micro and macro (process) perspectives

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Acknowledging partners...


Univ of Alicante


UFSC and Univ of Porto


INFAP - UNSL

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