Strategies for efficient He trapping

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OVERVIEW

ADDENDUM FCC-GOV-CC-0143: gas adsorbing materials and their applications as **residual gas trapping systems** in Ultra High Vacuum (UHV) systems of particle accelerators with focus on **He absorption** for the insulation vacuum.

- Gas Adsorption
- Materials
- Characterization
- Preliminary Results

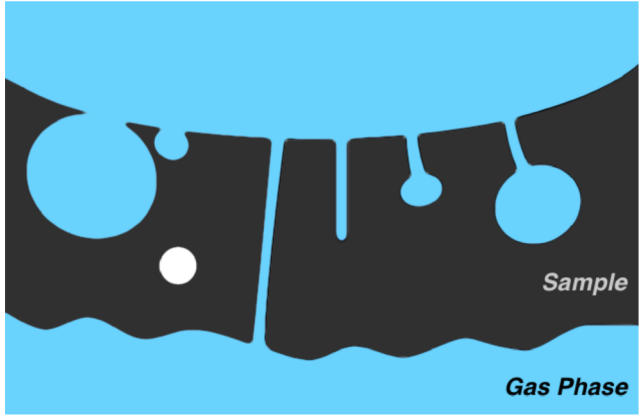
- PRESENT LHC CRYOSORBER RIBBON
- FILTERCARB GCC 8X30
- ACPC
- APAC

GAS ADSORPTION

Ultra-microporous materials

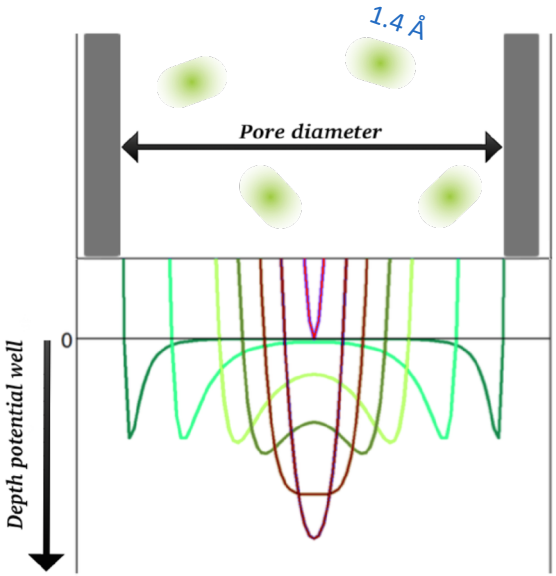
- High density of pores
- Internal pore surface >> external surface
- ★ Specific Surface Area > 1000 m^2/g

★ Ultra micropores *d < 1 nm*



van der Waals potential superposition

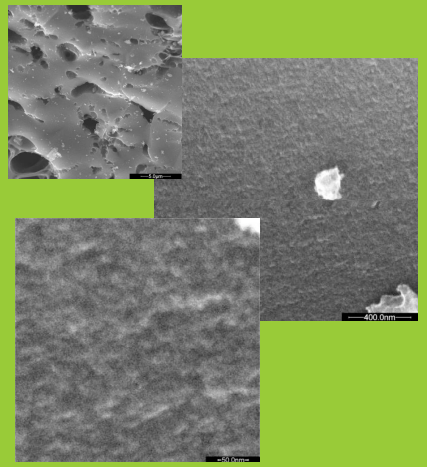
He vdW radius 1.40 Å



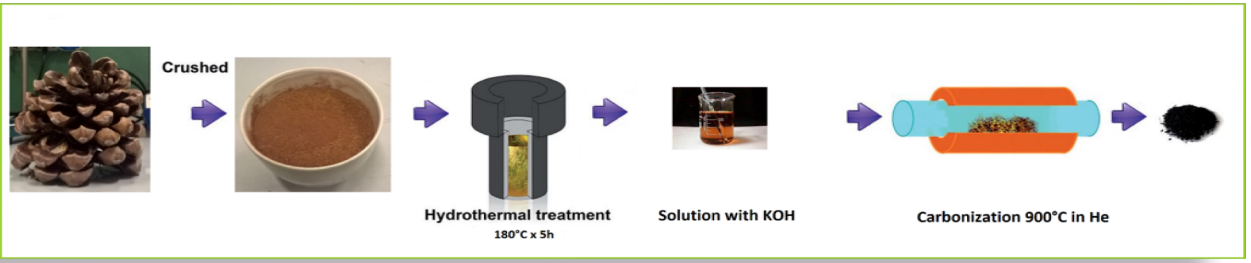
MATERIALS

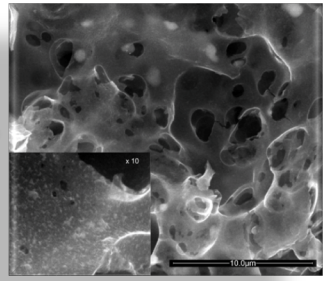
PRESENT LHC CRYOSORBER RIBBON

COMMERCIAL ACTIVATED CARBONS
FILTERCARB GCC 8X30



MATERIALS: ACPC Chemically Activated Carbons from Vegetal raw material

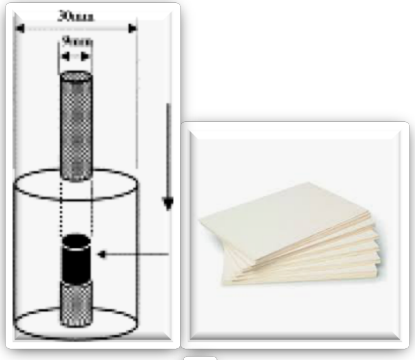




Production procedure: The samples were obtained by chemical activation of **pinecones** from Stone Pine (*Pinus pinea*), collected from the local area of Sila, Italy, with next steps:

- washed with distilled water to remove soluble impurities and dried at 353K for 12h;
- pulverized using a blender and heated at 453K for 5h to remove the undesirable moisture;
- placed in a solution (100 ml) of demineralized water with a weight ratios of KOH/DPC = 1 and stirred for 24h at 353K and then dried at 373K;
- transferred to a ceramic crucible in a tubular furnace under He with flow of 100 ml/min and heated at 1173K for 2h with a heating rate of 5K/min.

MATERIALS: APAC Physically Activated Carbons from amorphous Cellulose



Production procedure: The samples were obtained by physical activation of **commercial amorphous cellulose**, with next steps:

- crashed;
- pressed with mechanical press to obtain a pellet;
- transferred to a ceramic crucible in a tubular furnace under CO_2 with flow of 100 ml/min and heated at 1173K for 1h with a heating rate of 5K/min.

Solid ac-C bricks: shapeable adsorbers

CHARACTERIZATION

TEXTURAL PROPERTIES

- Textural properties were carried out by N_2 physical adsorption at 77K using a Micromeritics ASAP 2460 apparatus.

Before each adsorption measurements, the activated samples were dried under vacuum

He ADSORPTION

- The **He adsorption measurements**, at **77K and RT**, were carried out in the pressure range 0-1 bar by using a Micromeritics ASAP 2460 apparatus.

The gas storage capacity was determined from adsorption isotherms. Prior to each

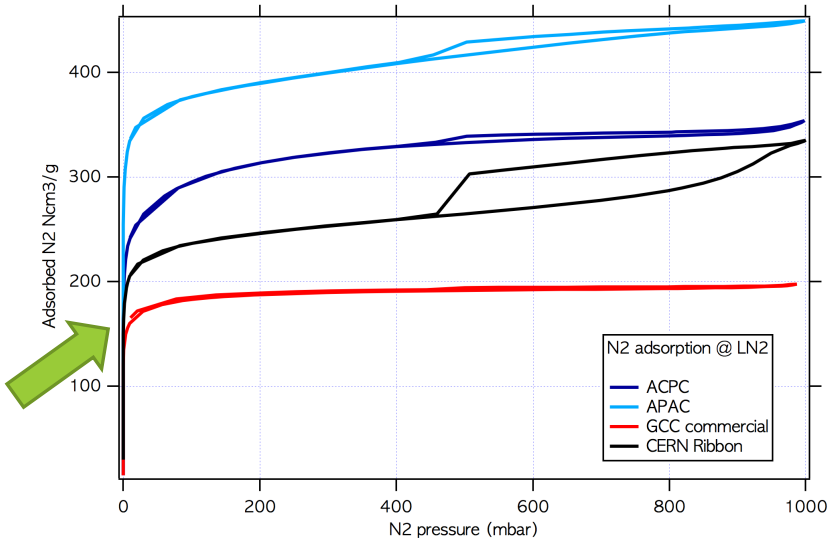
- The **Specific surface area** - SSA was calculated according to the **Brunauer-Emmett-Teller (BET)** method (1) within relative pressure range of 0.005 - 0.11.
- The **total pore volume V_T** was calculated from the N_2 uptake at relative pressure P/P_0 of 0.995.
- The **total micropore volume V_{micro}** and the **pore size distributions (PSD)** were calculated by **Density Functional Theory (DFT)** considering slit-shaped pores (2).

Micromeritics ASAP 2460 apparatus

1. Adsorption of gases in multimolecular. S. Brunauer, P.H. Emmett, E. Teller. 1938, J. Am. Chem. Soc. 60, 309. 38.
2. A new analysis method for the determination of the pore-size distribution of porous carbons from nitrogen adsorption measurements. N.A. Seaton, J. Walton, N. Quirke. 1989, Carbon 27, 853-861.

PRELIMINARY RESULTS: Textural Properties

Nitrogen adsorption-desorption isotherm at 77K

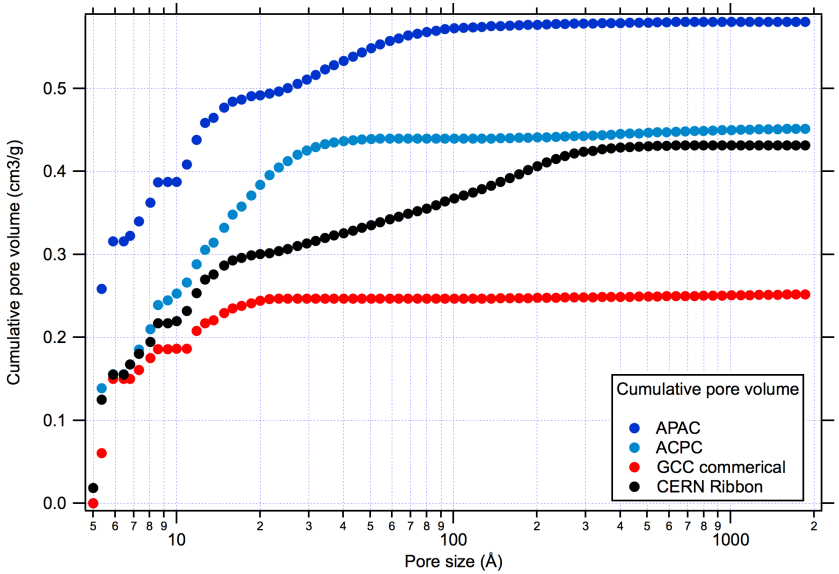


- All the samples show Type I and Type IV isotherm according to the IUPAC classification [3], thus they are microporous materials with developed mesopores.
- All isotherms show a **sharp increase of N2 uptake** at very low relative pressure (P/P0 < 0.01), symptomatic of **their microprous character**; a quasi-horizontal plateau in the range P/P0 > 0.1, whose values is strictly related to the micropore capacity, and a hysteresis loop at the relative pressure range of 0.4 - 1.0, associated with the capillary condensation taking place in mesopores.

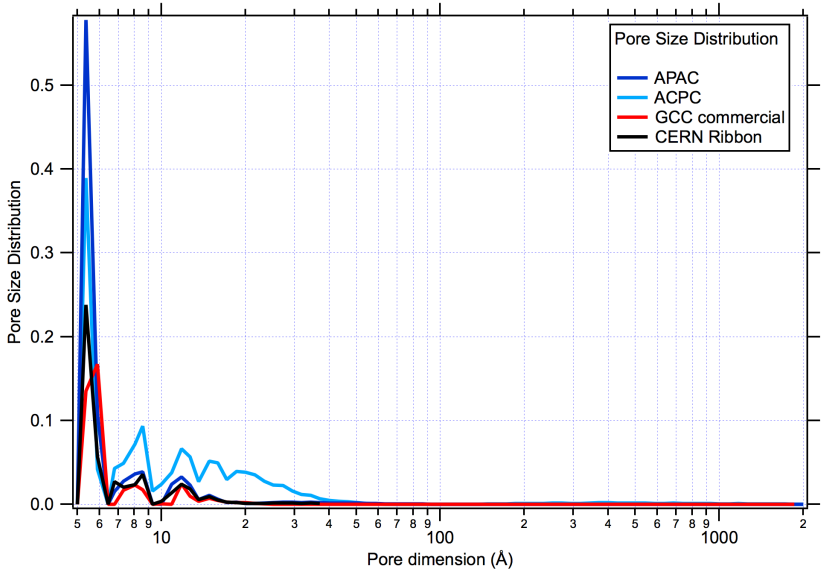
3. K.S.W. Sing, D.H. Everett, L. Haul, R.A. Pierotti, J. Rouquerol, T. Siemieniewska. Reporting Physisorption Data for Gas/Solid Systems, Handbook of Heterogeneous Catalysis. s.l. : Wiley-VCH Verlag GmbH & Co., KGaA, 2008.

PRELIMINARY RESULTS: Textural Properties

Cumulative Pore Volume



Pore size distribution



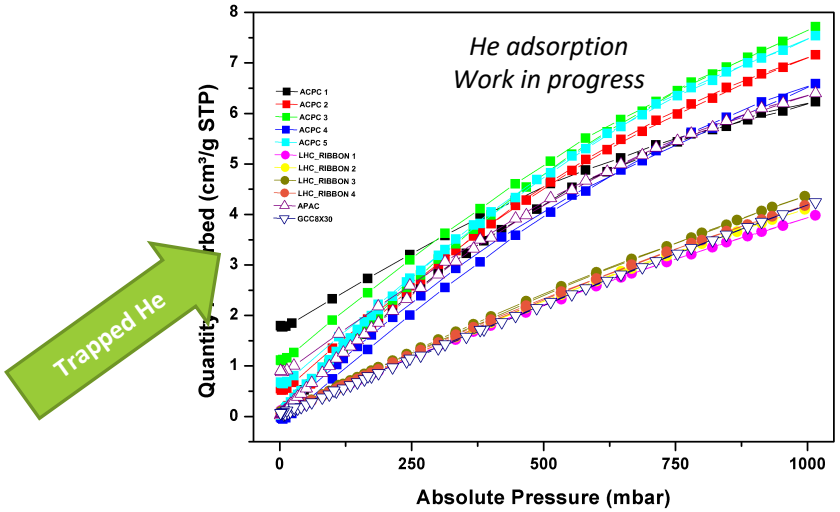
PRELIMINARY RESULTS: Textural Properties

SAMPLE	[a]S_{BET} (m^2/g)	[b]V_T (cm^3/g)	[c]V_{micro} (cm^3/g)	DFT cumulative pore volume (cm^3/g)		[f]V_{meso} (cm^3/g)	[g]V_{macro} (cm^3/g)	[h]F_{micro} (%)	[i]He (cm3/g STP)	[l]He (cm3/g STP)
				[d]Ultra - micropores	[e]Super - micropores					
ACPC	**1200**	0.4513	0,3836	0.1854	**0.1982**	0.0630	0.0048	85	7	0.7
LHC_RIBBON	942	0.4313	0,3002	0.1800	0.1202	0.1303	0.0008	70	4,2	0,2
APAC	**1500**	0.5803	0.4918	0.3397	**0.1521**	0.0877	0.0008	85	6,4	0,4
GCC8X30	726	0.25182	0.24399	0.1606	0.08339	0.00255	0.00528	97	4,24	0

[a] SBET: specific surface area computed using BET equation in the relative pressure range of 0.005–0.1.
[b] V_T: total pore volume estimated at a relative pressure $P/P_0 = 0.99$.
[c] V_{micro}: micropore volume determined from cumulative volume.
[d] Ultramipores: Volume of pores with less than 0.7 nm width
[e] Supermicropores: Volume of pores with less than 2 nm width
[f] V_{meso}: Volume of pores with less than 50 nm width.
[g] V_{macro}: macropore volume determined from subtraction of micropore and mesopore volume from total pore volume.
*[h] F_{micro}: fraction of micropore volume = (micropore volume/total pore volume) * 100*
[i] He uptake: hydrogen storage capacity at 77 K and 1 bar.
[l] He uptake: hydrogen storage capacity at RT and 1 bar

PRELIMINARY RESULTS: He Adsorption

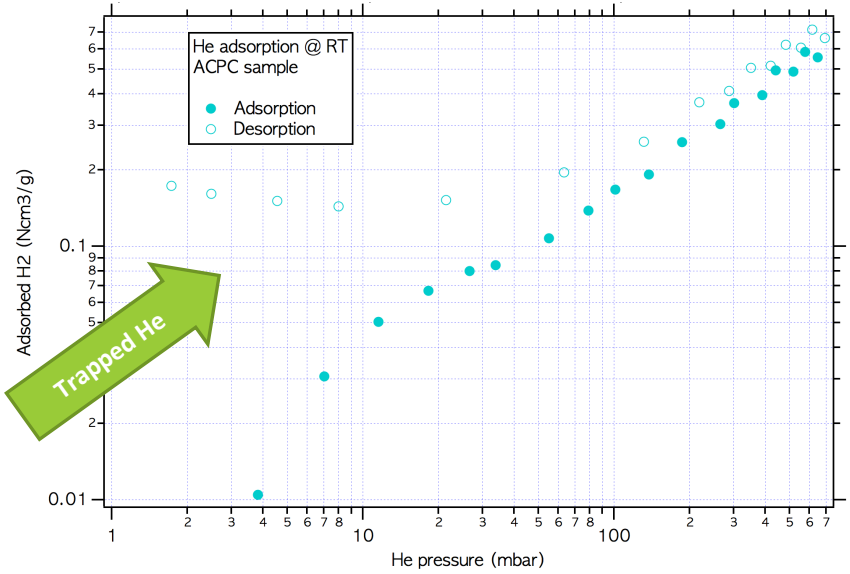
He Adsorption-Desorption Isotherm at 77K



- **+ 75% He adsorption:** 77K isotherms show a maximum storage capacity at 1 bar that increases from a minimum value around ~ 4 cm^3/g for the LHC_RIBBON and GCC 8X30 sample to a maximum value close to **~ 7 cm^3/g in the APAC and in ACPC**.
- **Full reactivation:** Cycles following the first lead to a slight change in the maximum adsorption capacity, which could easily be attributed to instrumental error that could be easily recovered by a mild thermal treatment at 473K for 12 h.
- **He trapping:** The trapping of the He molecule by the pores suggest that the interaction between sorbent and adsorbate is due to a strong physisorption process that results almost completely reversible.

PRELIMINARY RESULTS: He Adsorption

He Adsorption-Desorption Isotherm at RT



- **Effective potentials:** ACPC and APAC adsorb He at RT (+300% with respect to CERN Ribbon)
- **He trapping at RT**
- The GCC does not show He adsorption at RT

SAMPLE	He (cm3/g STP)		He (mmol/g)		He (gHe/g)		He (WT%)	
	77	RT	77	RT	77	RT	77	RT
ACPC	7,0	0,7	0,31	0,031	1,25	0,125	0,125	0,012
LHC_RIBBON	4,2	0,2	0,19	0,009	0,75	0,036	0,075	0,004
APAC	6,4	0,4	0,29	0,018	1,14	0,071	0,114	0,007
GCC8X30	4,2	0	0,19	0,000	0,76	0,000	0,076	0,000

4. J. Toth, Adsorption: Theory, Modeling, and Analysis. New York: Marcel Dekker, 2002

PRELIMINARY CONCLUSIONS

1. Shapeable C-based He adsorbers
2. Enhanced He adsorption
3. Full reactivation
4. He trapping phenomenon

Outlooks

1. Optimization of the textural properties
2. Test in operative conditions
3. Production of He adsorber units