Università degli Studi di Salerno

Dipartimento di Chimica e Biologia

Aerogeli Polimerici

Dottorato in Chimica
XII ciclo

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Monolithic aerogels

Solid materials that are so porous that they contain mostly air (the pores occupy a very high percentage of a sample volume, above 90%).

- ✓ Inorganic Aerogels
- ✓ Chemically crosslinked polymeric aerogels
- ✓ Carbon Aerogels
- ✓ Physically crosslinked polymeric aerogels

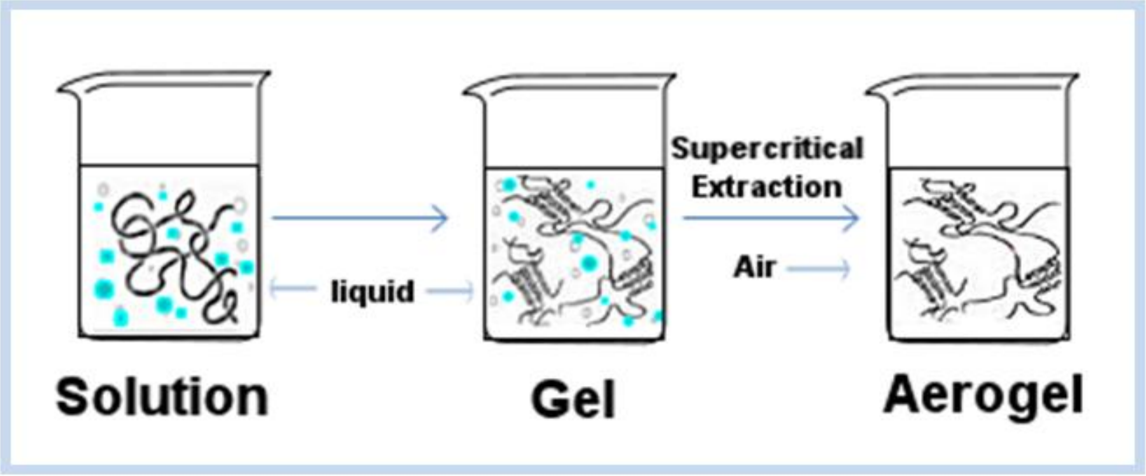
H.D,Gesser; P.C.,Goswami;*Chem.Rev* **1989**,89,765-788

Monolithic physically crosslinked polymeric aerogels

Physical Gels

- Polymers should be →
  - High MM (preferentially)
  - **Regular and stereoregular(stable crystalline phases)**

- **Hot polymer solutions are sudden cooled to lower temperature where gelation occurs.**



High temperature solution

Low temperature physical gel

Gels and Aerogels where links between chains are crystalline regions

Monolithic physically crosslinked polymeric aerogels

Monolithic physically crosslinked polymeric aerogels based on thermoplastic commercial polymers :

- Isotactic poly(4-methyl-pentene-1) i-P4MP1
- Ultra High Molecular Weight Polyethylene UHMWPE

Monolithic physically crosslinked polymeric aerogels based on thermoplastic polymers with nanoporous crystalline phase :

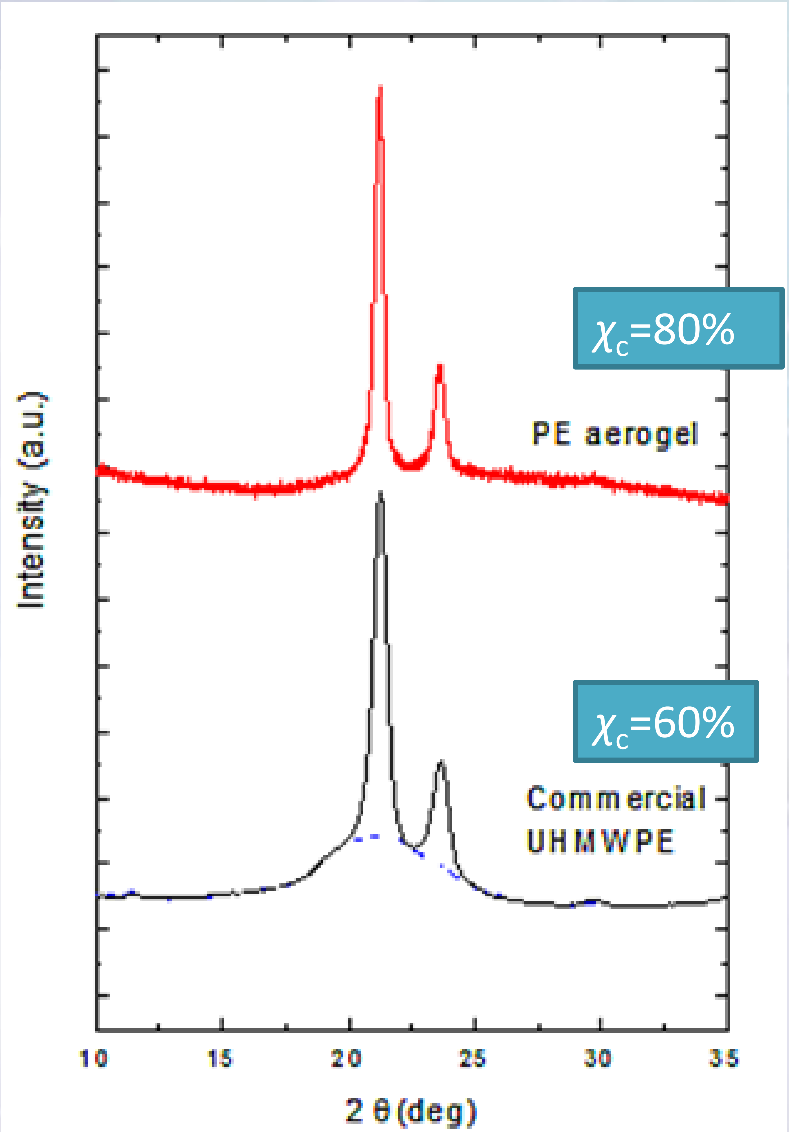
- Syndiotactic polystyrene (s-PS)
- Aerogels based on syndiotactic polystyrene and poly(2,6- dimethyl-1,4-phenylenoxide) PPO
- Aerogels based on syndiotactic polystyrene and poly(2,6-diphenyl-1,4-phenylenoxide) PPPO

Nanocomposites Physically Crosslinked Polymeric Aerogels based on s-PS with :

- Organically Modified Montmorillonite (OMMT)
- Reduced Graphene Oxide (r-GO)

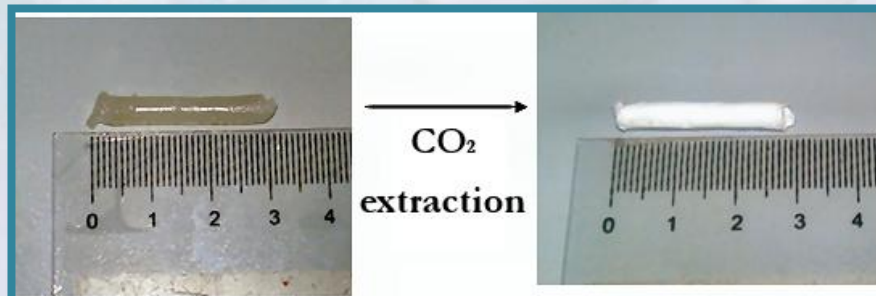
Monolithic physically crosslinked polymeric aerogels based on thermoplastic commercial polymers

Polyethylene Aerogels

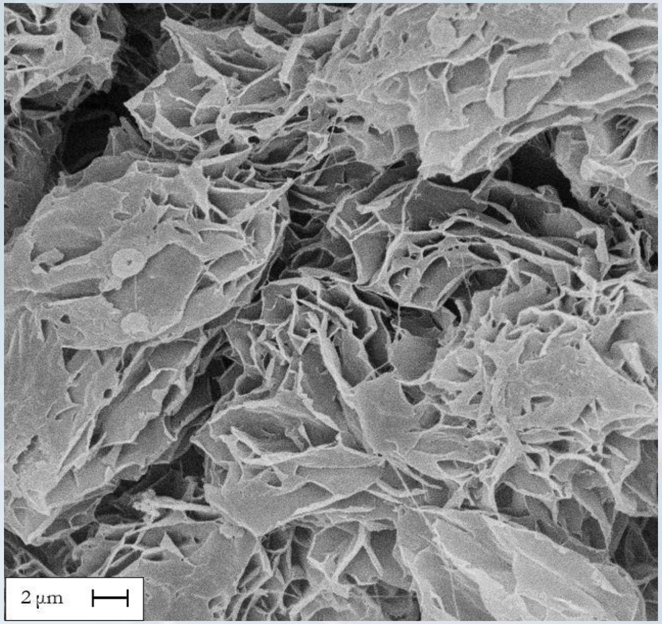


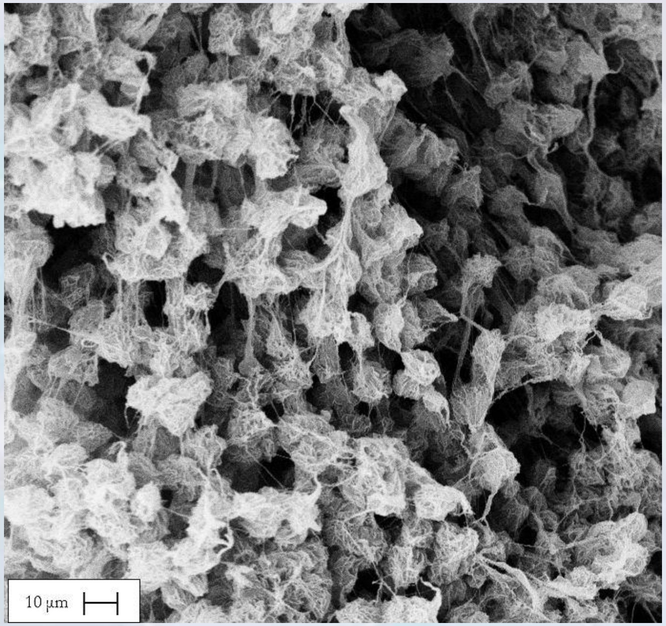
- 10 wt% of polymer (solv.decaline)
- 0.5 % w/w of the anti-oxidant di-t-butyt-p-cresol

P. Smith, P. J. Lemstra, H. C. Booij,J. Polym. Sci., Phys. ,**1981**, Ed. 19, 877



Polyethylene Aerogels





Sample	S_{BET} (m^2 g^{-1})
Commercial UHMWPE	2,7
UHMWPE aerogel	66

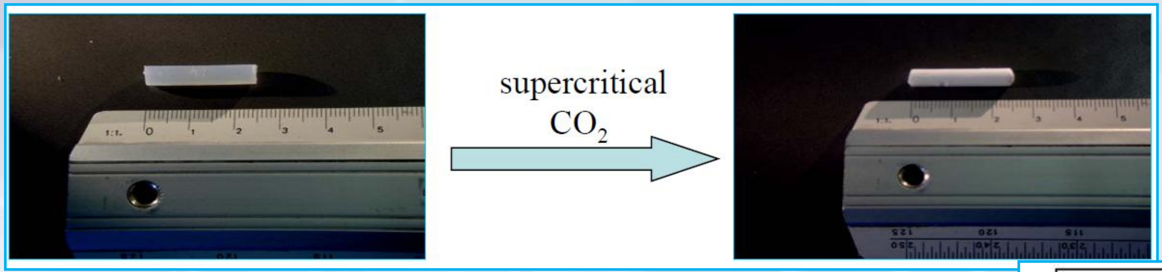
Total area evaluated following the BET model in the standard $0.05<P/P_0<0.3$ pressure range.

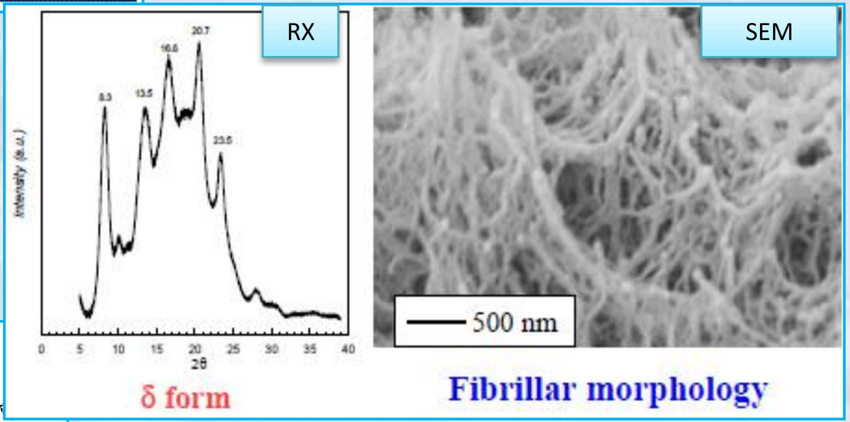
Monolithic physically crosslinked polymeric aerogels based on thermoplastic polymers with nanoporous crystalline phase

Polymeric aerogels with nanoporous crystalline phase

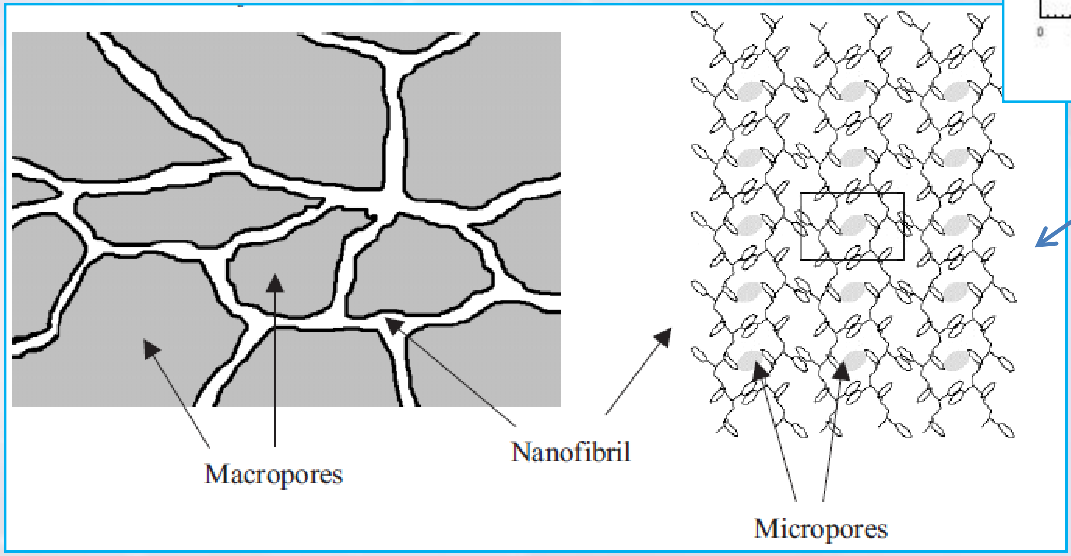
Monolithic physically crosslinked polimeric aerogels

sPS aerogel





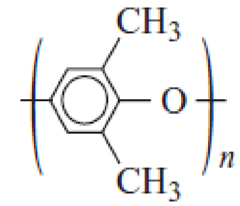
crystalline regions consist of a clathrate phase



C. Daniel,D. Alfano,V. Venditto,S. Cardea,E. Reverchon, D. Larobina, G. Mensitieri, G.Guerra *Advanced Material*,**2005,**17(12),1515-1518.

Monolithic Nanoporous Crystalline Aerogels based on PPO

Preparation of monolithic aerogels, including PPO nanoporous-crystalline phases

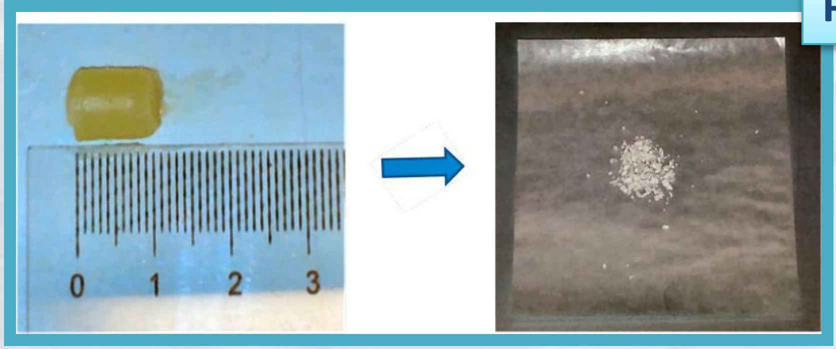


PROBLEM :

solvent extraction by supercritical CO_2 from PPO gels leads to powders rather than to aerogels

Daniel, C.; Longo, S.;Vitillo, J.G.;Fasano, G.;Guerra, G. *Chem. Mater*. **2011**, *23*, 3195

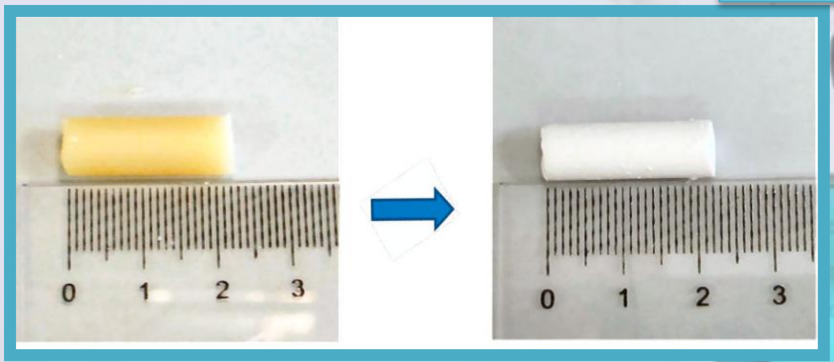
PPO



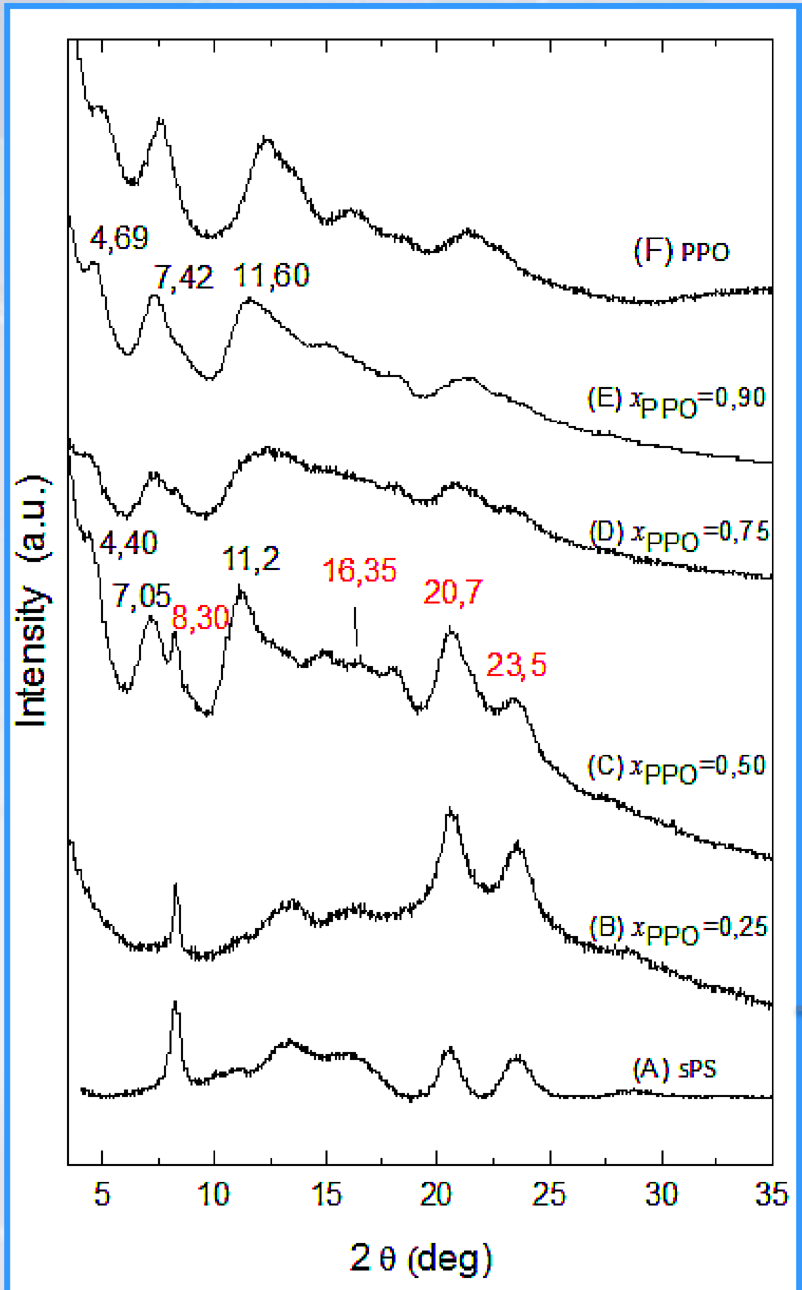
SOLUTION:

s-PS/PPO aerogels

PPO 90wt%
sPS 10wt%



Influence of s-PS on PPO crystallization



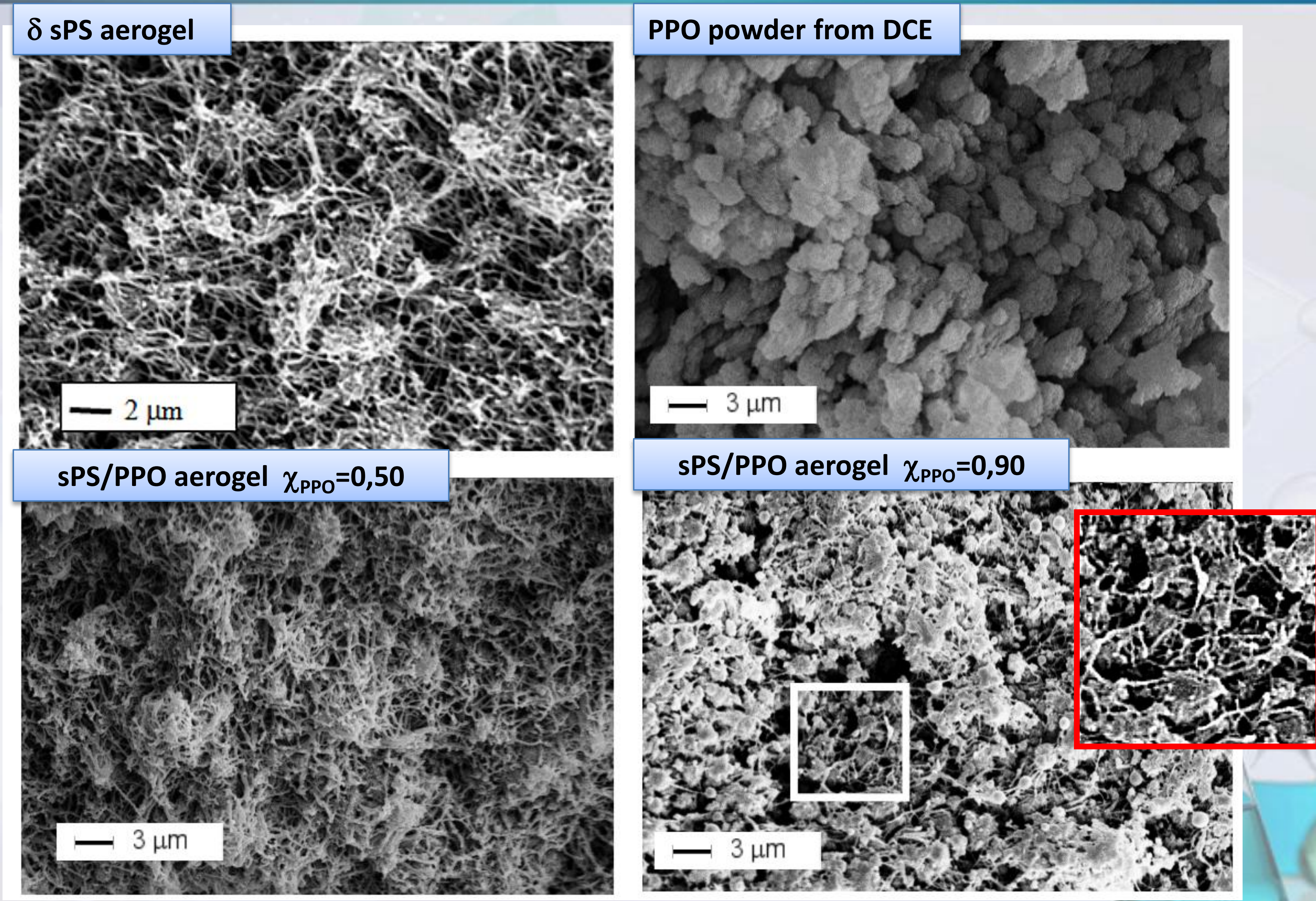
PPO crystallinity diffraction peak positions change with χ_{PPO}

Both s-PS δ form and PPO crystallinities are clearly present

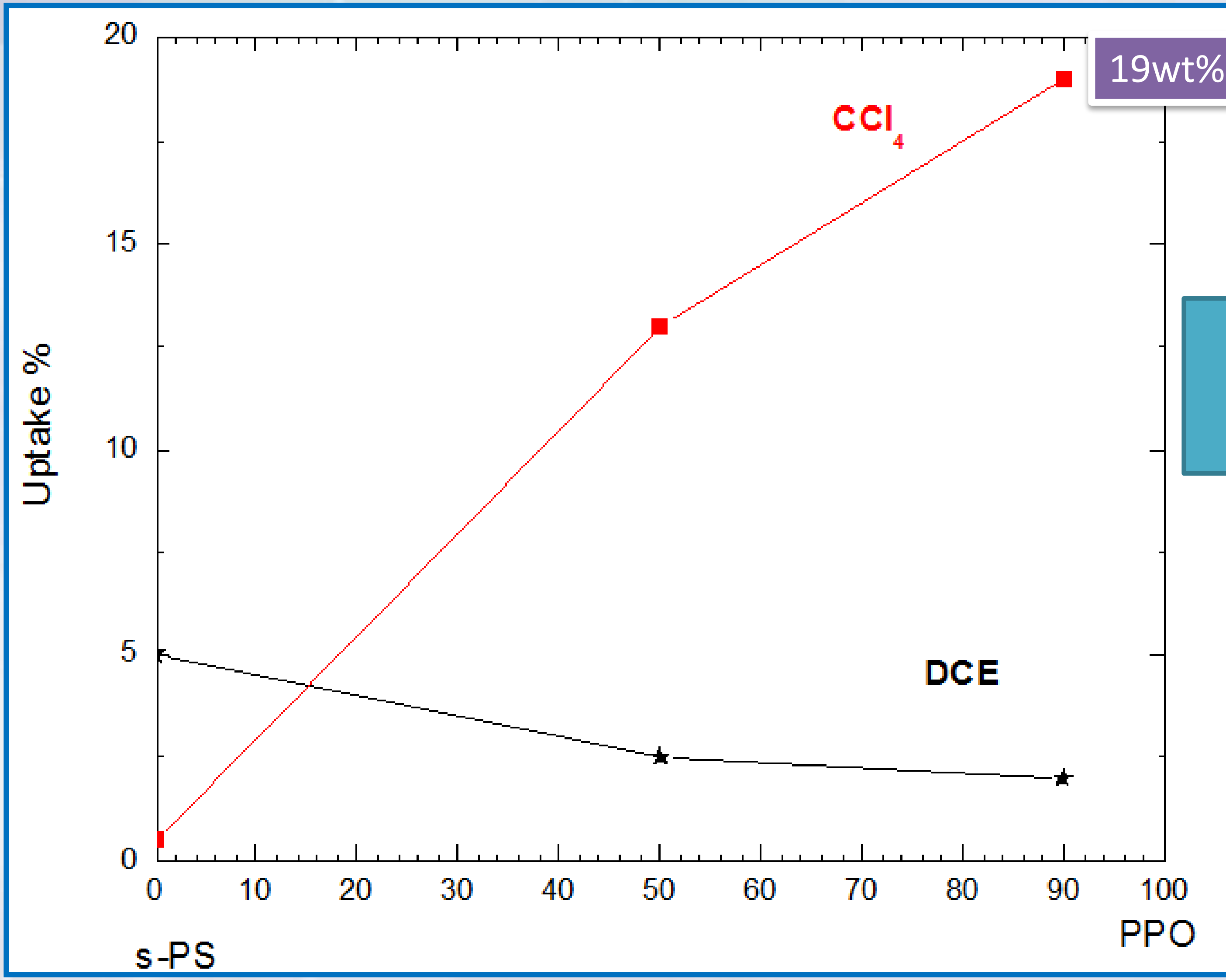
For low PPO contents only the crystallinity of the δ form of s-PS, is present.

Daniel, C.; Longo, S.;Vitillo, J.G.;Fasano, G.;Guerra, G. *Chem. Mater*. **2011**, *23*, 3195

Influence of s-PS on PPO crystallization



Sorption from diluted aqueous solutions

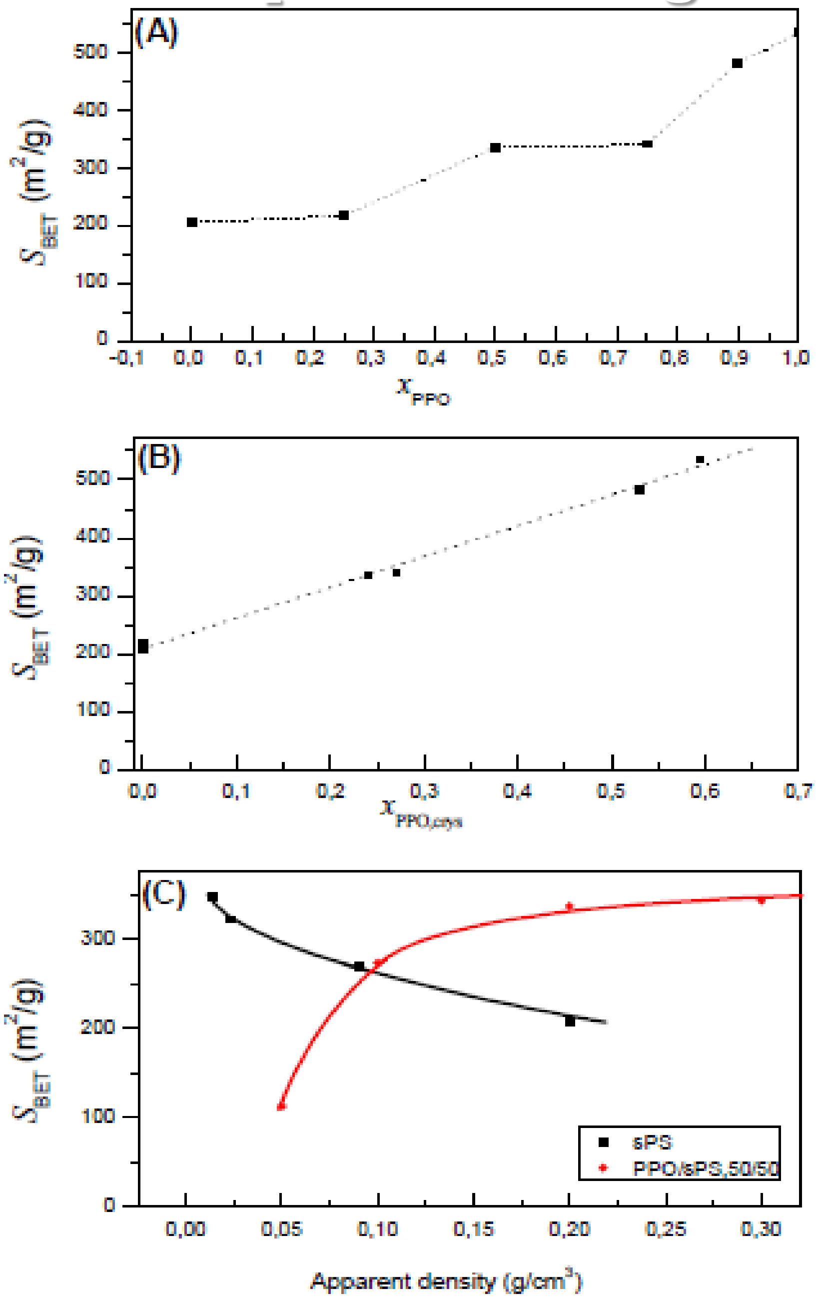


19wt%

Selective Absorption

10 ppm

Sorption Properties:
Properties of aerogels based on nanoporous-crystalline PPO

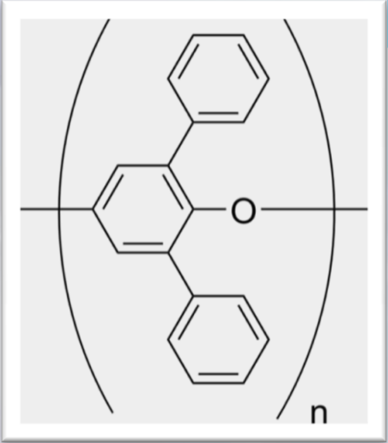


The surface area , $S_{BET,}$ increases with the PPO content but only for aerogels with crystalline PPO

The surface area increases when the density increases

Total surface areas and micropore areas, as obtained from N_2 isotherms (BET experiments) for the mixed PPO/s-PS aerogels

Monolithic aerogels based on PPPO



Poly(2,6-diphenyl-1,4-phenylene oxide)

High sorbent nanoporous amorphous phase

- **Thermally stable (up to 350 °C)**

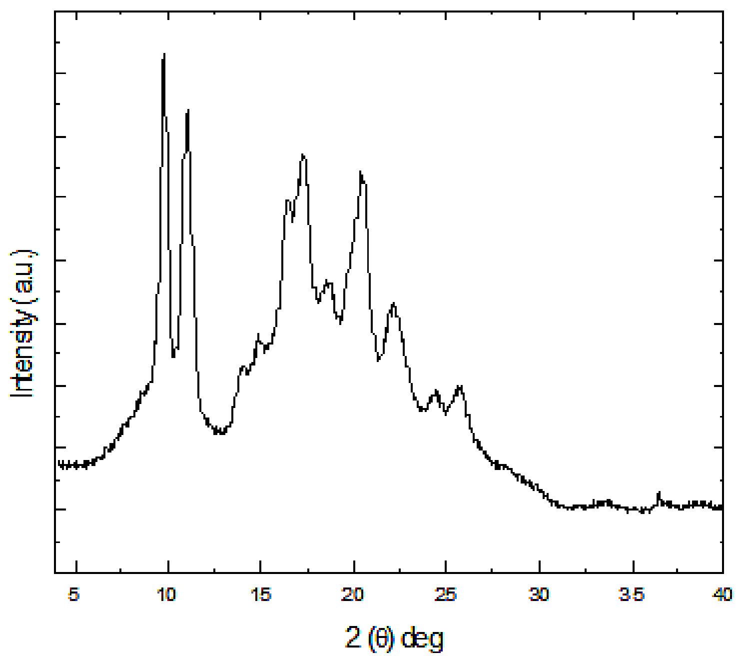
K. Dettmer, W. Engewald, *Anal. Bioanal. Chem.* **2002**, 373 (6) ,490–500

- **High glass transition temperature (T_g=220°C)**

I. Maier, M. Fieber,J. High. Resolut. Chromatogr. **1988**,11 (8), 566–576

- **An adsorbent material for monitoring air or water quality and industrial emissions**

W. Bertsch, A. Zlatkis, H.M. Liebich, *J. Chromatogr.* **1974**, A 99, 673–687
M. Harper, *J.Chromatogr.* **2000**, A 885 (1-2), 129–151



Commercial Tenax TA

Monolithic aerogels based on PPPO

Main Goals

- Establish if the occurence of a nanoporous crystalline phase could be at the origin of the high sorption properties of this polymer.

- Explore the possibility to obtain PPPO aerogels, in the attempt to improve the already high sorption capability.

Monolithic aerogels based on PPPO

PPPO is not
able
to give thermoreversible
gels

↓

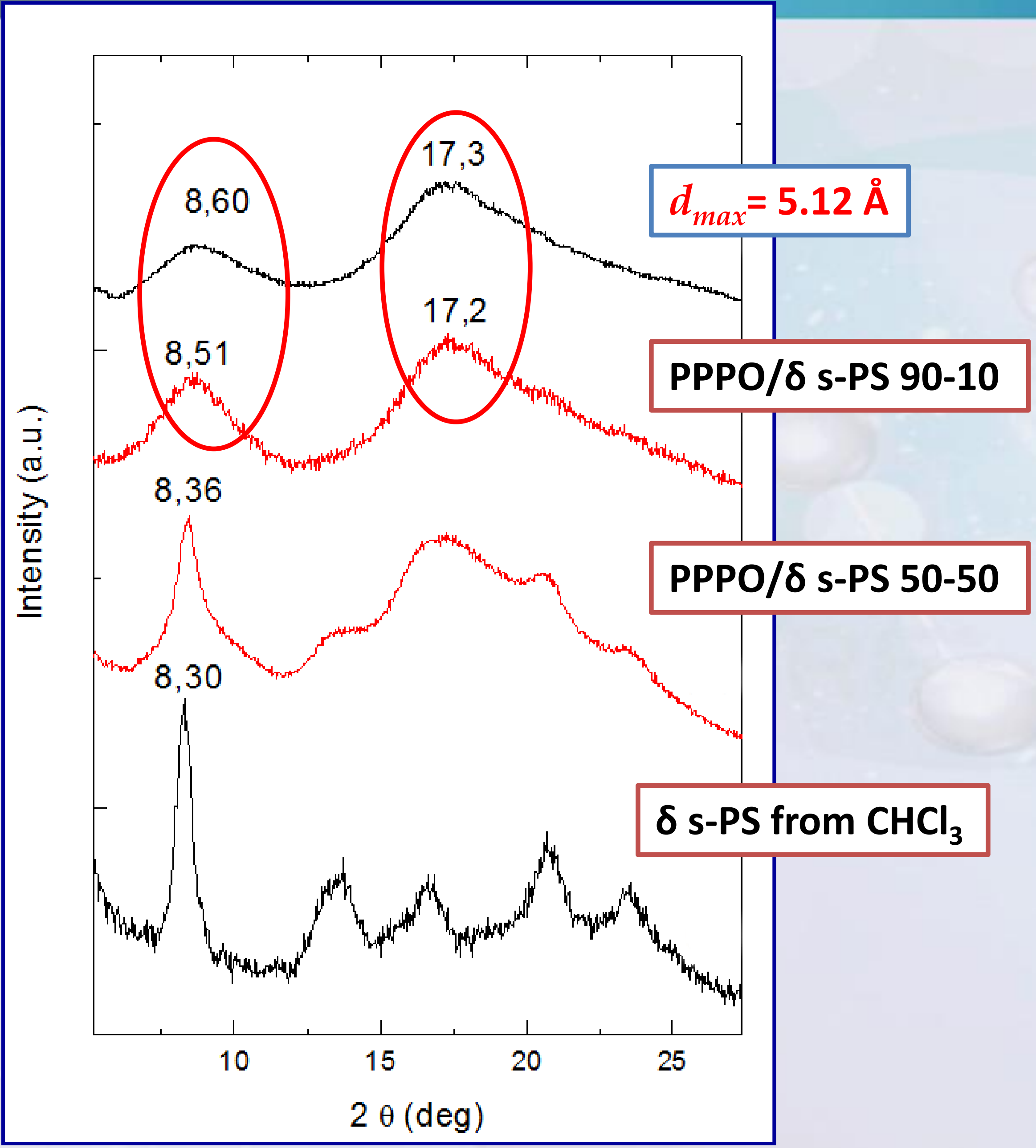
IT'S IMPOSSIBILE TO OBTAIN
AEROGELS

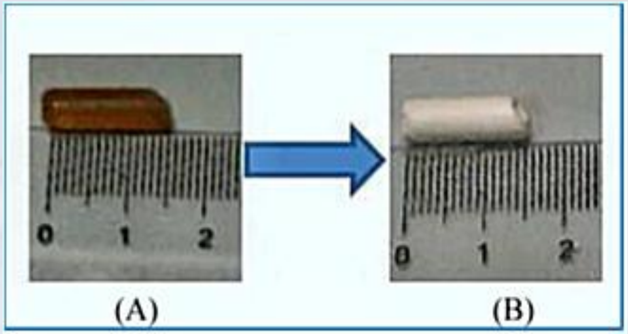
↓

BUT

Monolithic PPPO aerogels can be prepared from thermoreversible gels based on PPPO/syndiotactic polystyrene (s-PS) blends

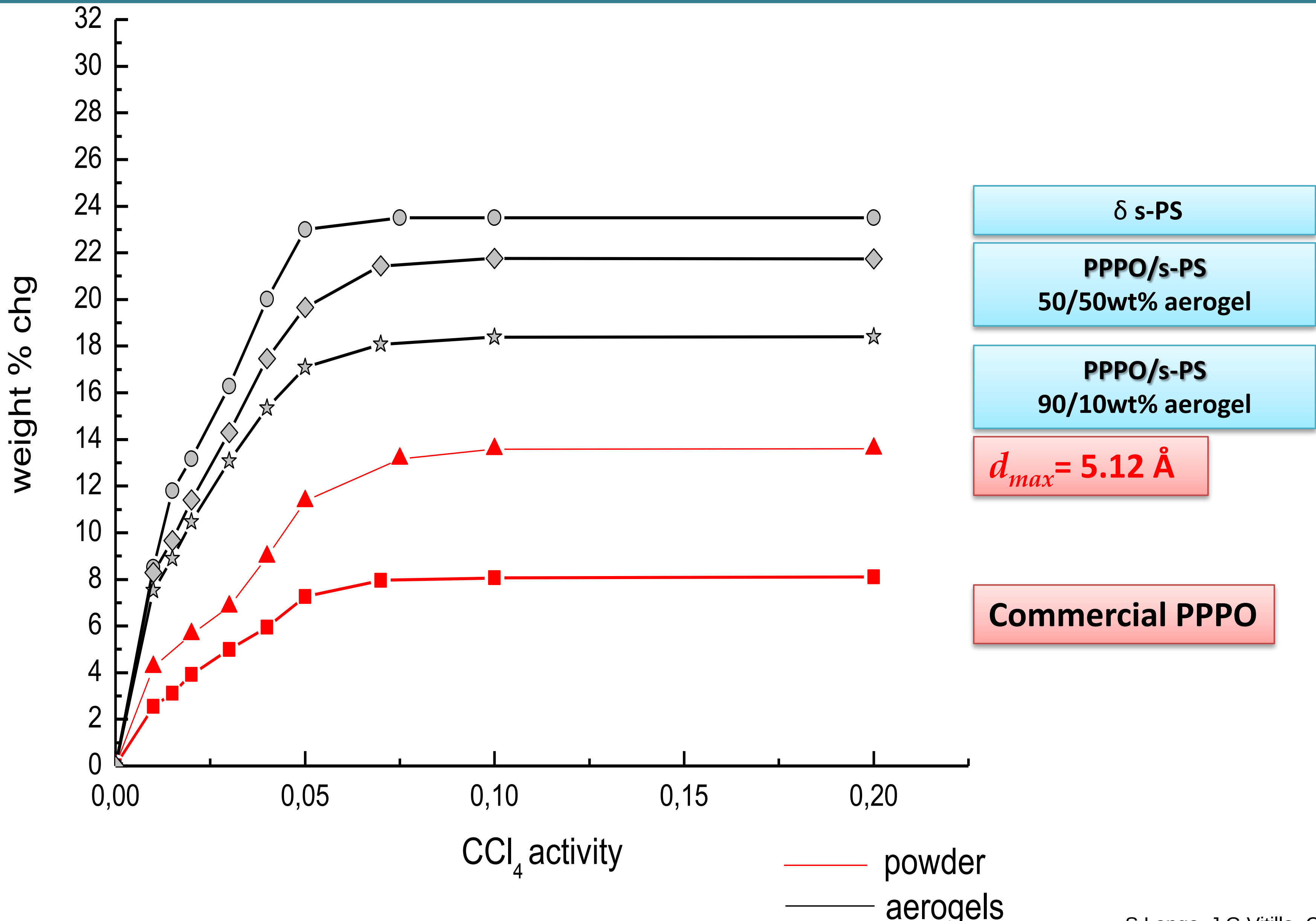
Monolithic aerogels based on PPPO





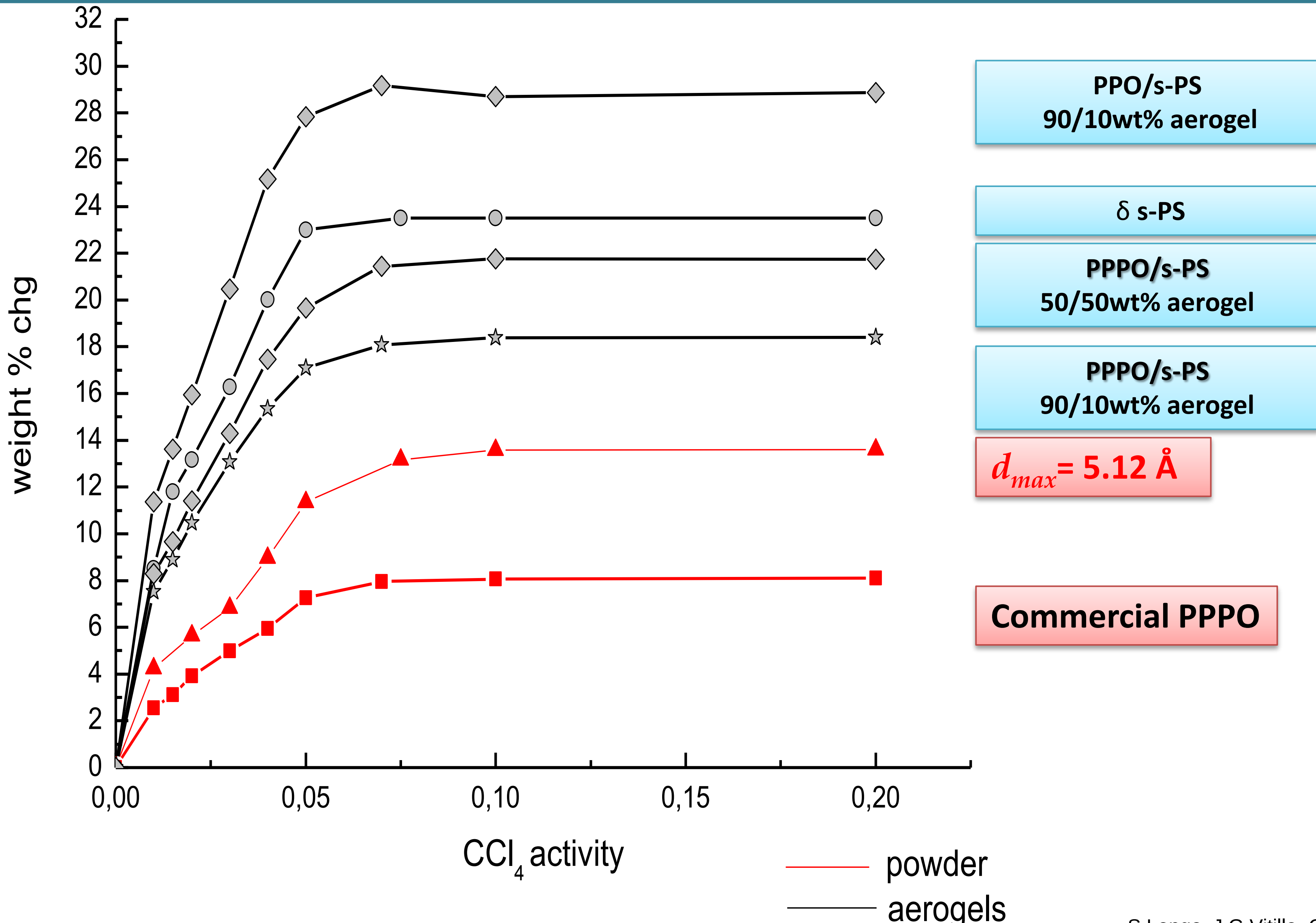
S.Longo, J.G.Vitillo, C.Daniel, G.Guerra, *ACS Applied Materials & Interfaces*, **2013**

Monolithic aerogels based on PPPO:sorption properties



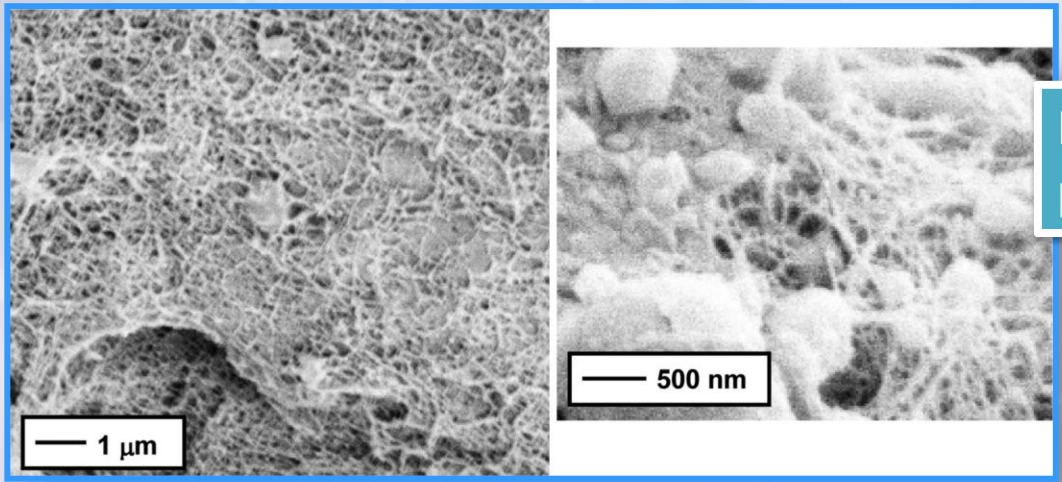
S.Longo, J.G.Vitillo, C.Daniel, G.Guerra, *ACS Applied Materials & Interfaces*, **2013**

Monolithic aerogels based on PPPO:sorption properties



S.Longo, J.G.Vitillo, C.Daniel, G.Guerra, *ACS Applied Materials & Interfaces*, **2013**

Monolithic aerogels based on PPPO



PPPO/s-PS aerogel with porosity P = 90%

Samples	Polymer composition (wt/wt)	S_{BET} ($m^2\ g^{-1}$)
Aerogels	s-PS(δ-form)	290
	PPPO/s-PS, 50/50	**197**
	PPPO/s-PS, 90/10	**47**
Powders	s-PS(δ-form)	43
	PPPO	**20**

BET measurements

Total area evaluated following the BET model in the standard $0.05<P/P_0<0.3$ pressure range.

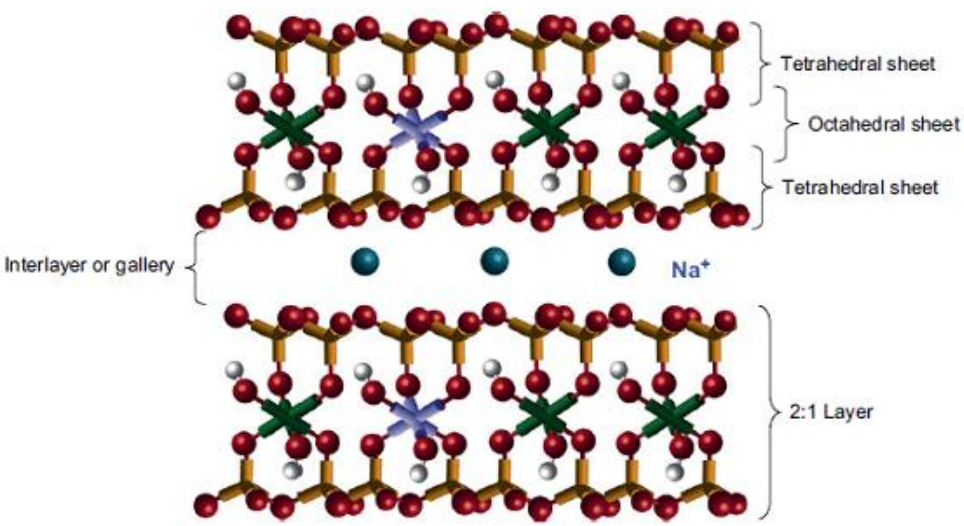
S.Longo, J.G.Vitillo, C.Daniel, G.Guerra, *ACS Applied Materials & Interfaces*, **2013**

Nanocomposites
Physically Crosslinked Polymeric Aerogels based on s-PS

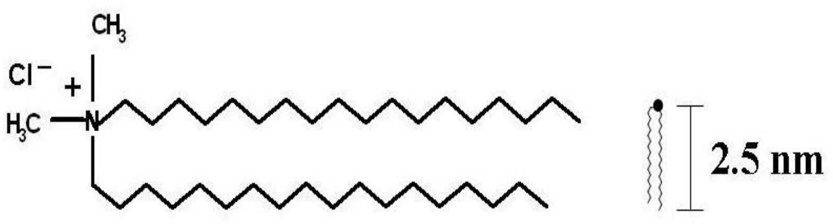
Nanocomposites Physically Crosslinked Polymeric Aerogels

Polymer/Clay Aerogels

Na^+–MMT **sodium montmorillonite**



OMMT :organically modified montmorillonite



2HT: Di(hydrogenated tallow) dimethyl ammonium chloride

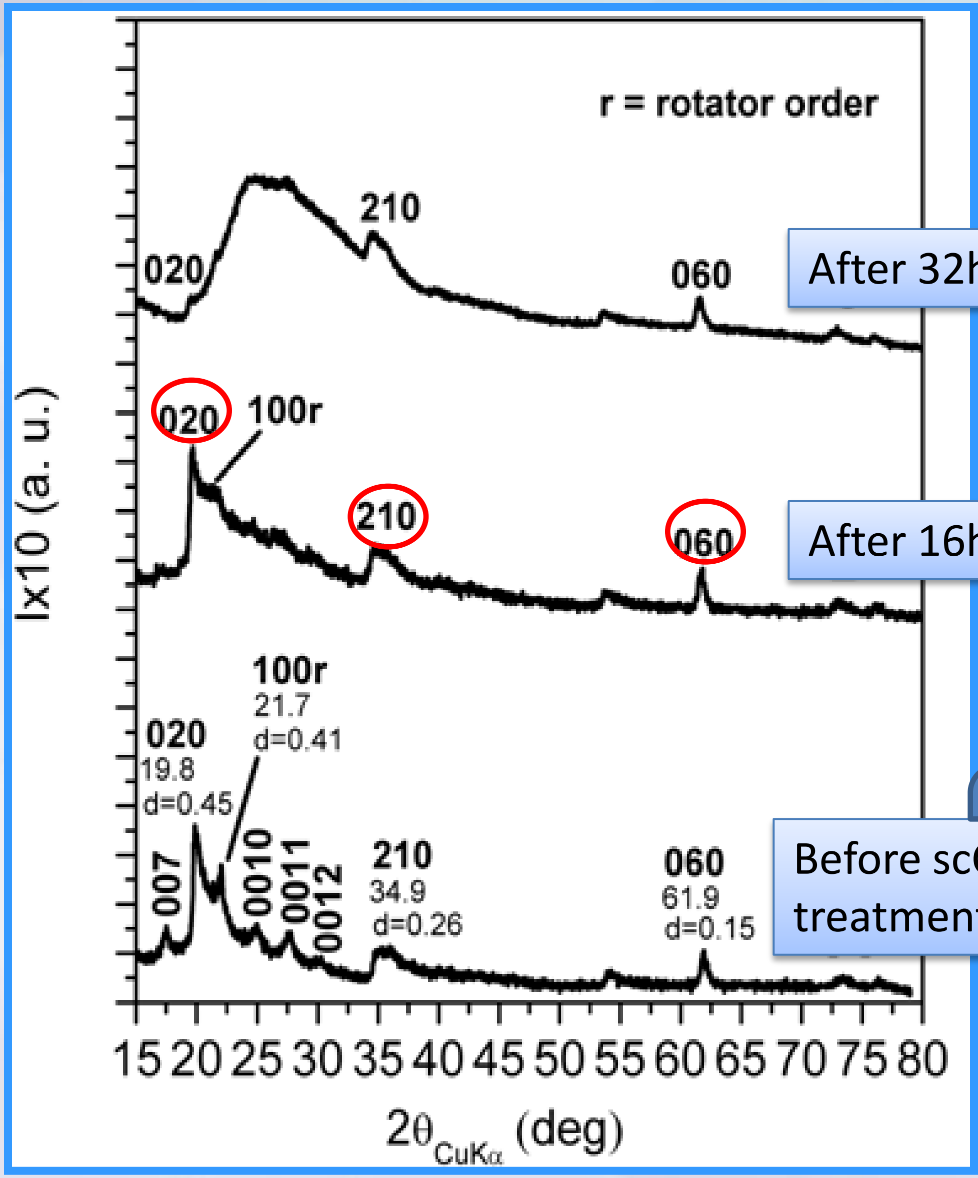
Main goals

- Investigation of the $scCO_2$ induced organoclay exfoliation
- Preparation of composite aerogels containing large amounts of exfoliated organoclay as well as a nanoporous-crystalline polymer phase.

Nanocomposites Physically Crosslinked Polymeric Aerogels

OMMT exfoliation by $scCO_2$

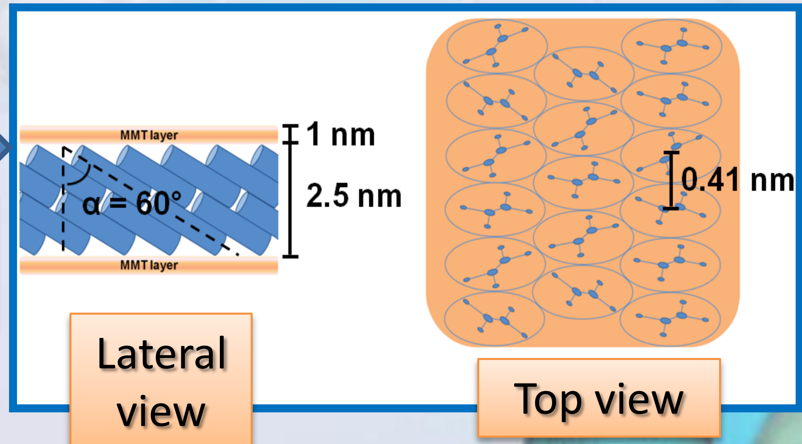
a)S. Horsch, G. Serhatkulu, E. Gulari, R. M. Kannan . *Polymer* , **2006**, 47, 7485–7496.
b) M. Manitiu, R. J. Bellair, S. Horsch, , E. Gulari, and R. M. Kannan, *Macromolecules* ,**2009**,41, 8038–8046.



After 32h $scCO_2$ treatment → Exfoliation is achieved

After 16h $scCO_2$ treatment → maintenance of the in-plane order

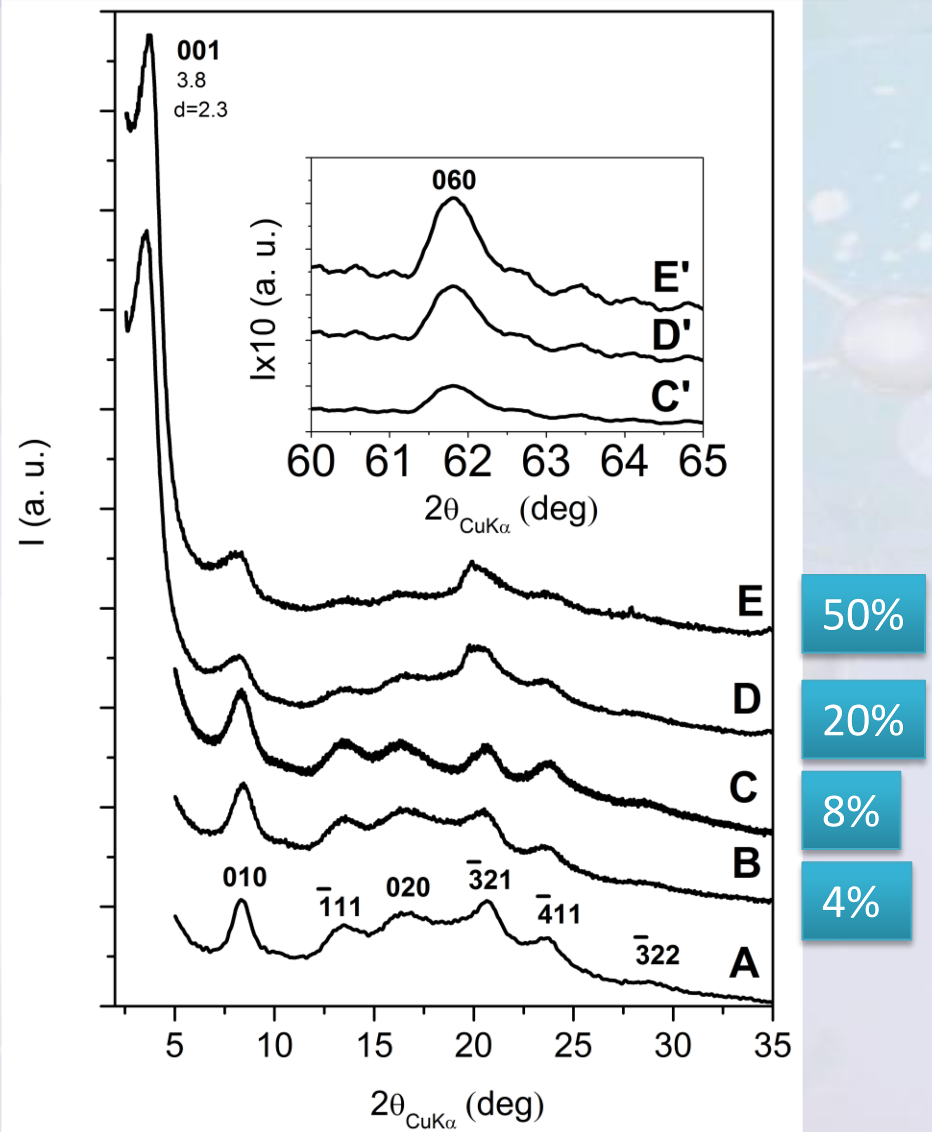
Before $scCO_2$ treatment



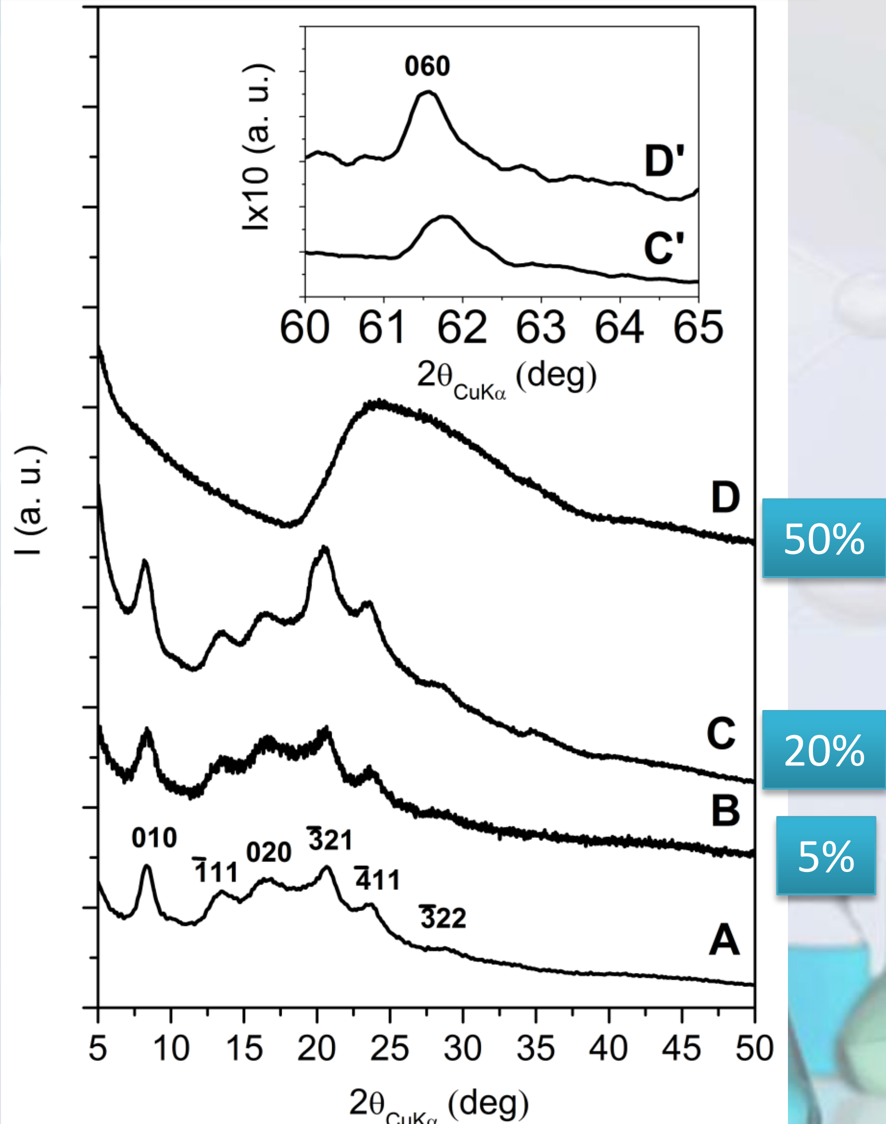
S.Longo,M. Mauro, C. Daniel, M. Galimberti, G. Guerra. Frontiers in Polymer Chemistry, ***2013***,*1,1-9.*

Nanocomposites Physically Crosslinked Polymeric Aerogels

s-PS aerogels with as received OMMT



s-PS aerogels with exfoliated OMMT

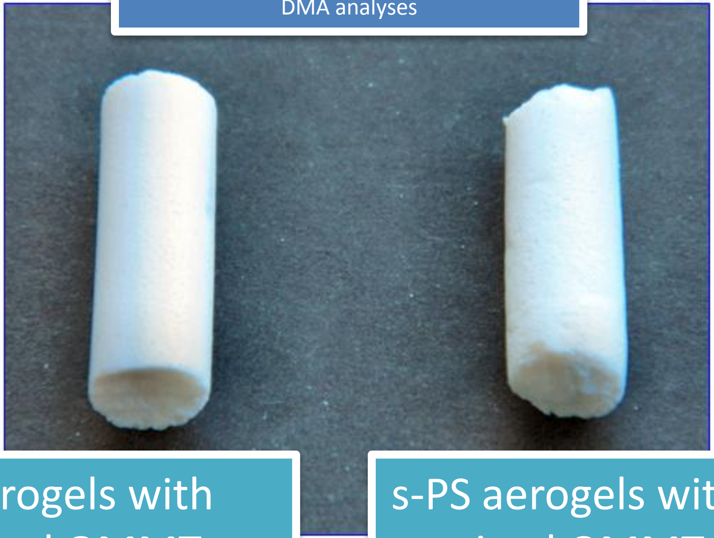


S.Longo, M. Mauro, C. Daniel, M. Galimberti, G. Guerra. Frontiers in Polymer Chemistry, ***2013,****1,1-9.*

Nanocomposites Physically Crosslinked Polymeric Aerogels

36 MPa vs 15 Mpa
DMA analyses

ELASTIC MODULUS



s-PS aerogels with exfoliated OMMT

s-PS aerogels with as received OMMT

80/20 weight ratio
P=90%

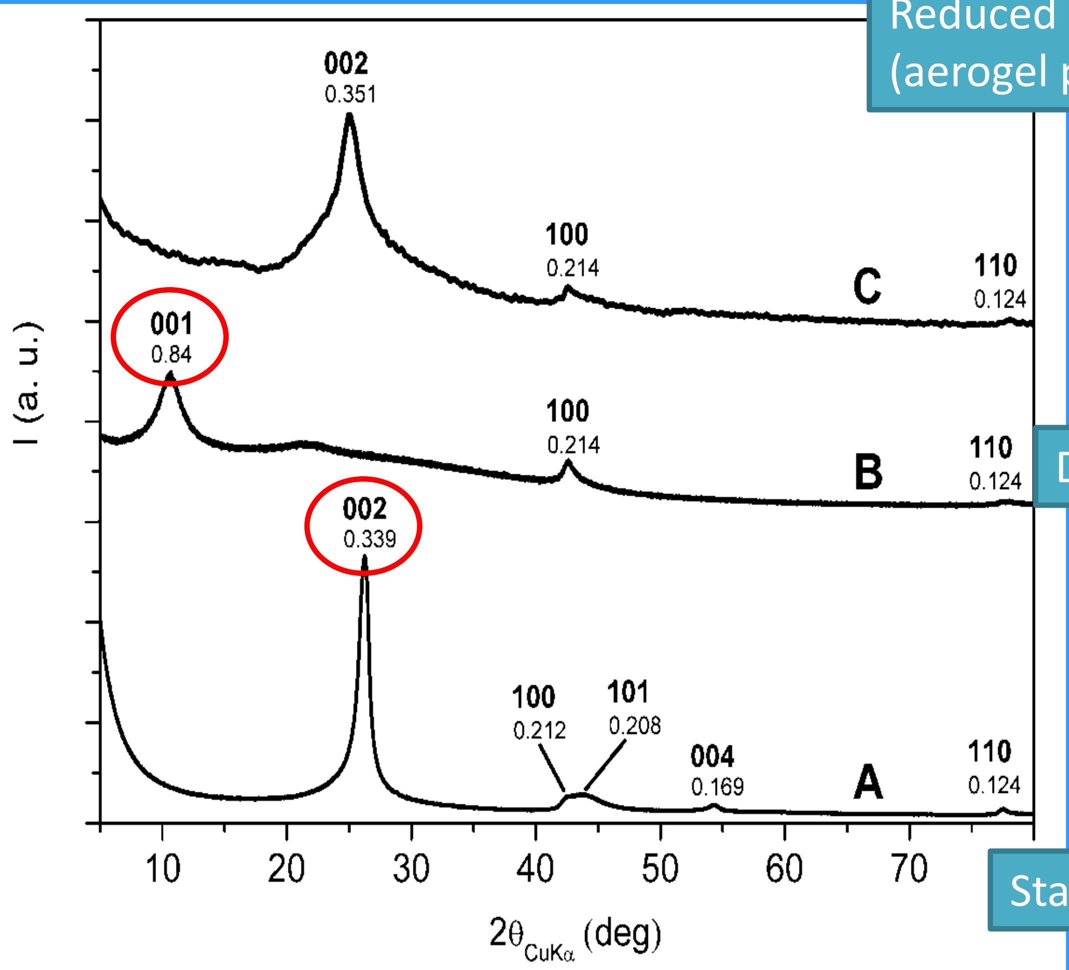
Sample	S_{BET}^{a} ($m^2\ g^{-1}$)
Intercalated OMMT	10
Exfoliated OMMT ($scCO_2$ treated)	18
s-PS/intercalated-OMMT, 80/20 aerogel	166
s-PS/exfoliated-OMMT, 80/20 aerogel	281
Aerogel δ s-PS	312

Total area evaluated following the BET model in the standard $0.05<P/P_0<0.3$ pressure range.

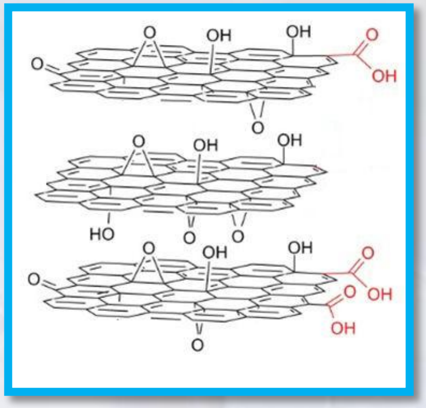
S.Longo,M. Mauro, C. Daniel, M. Galimberti, G. Guerra. Frontiers in Polymer Chemistry, ***2013,****1,1-9.*

Nanocomposites Physically Crosslinked Polymeric Aerogels

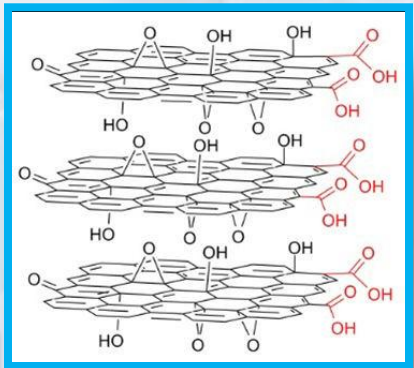
Polymer/GO Aerogels



Reduced GO
(aerogel preparations)



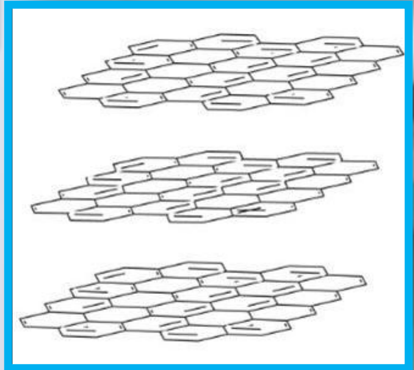
Derived GO



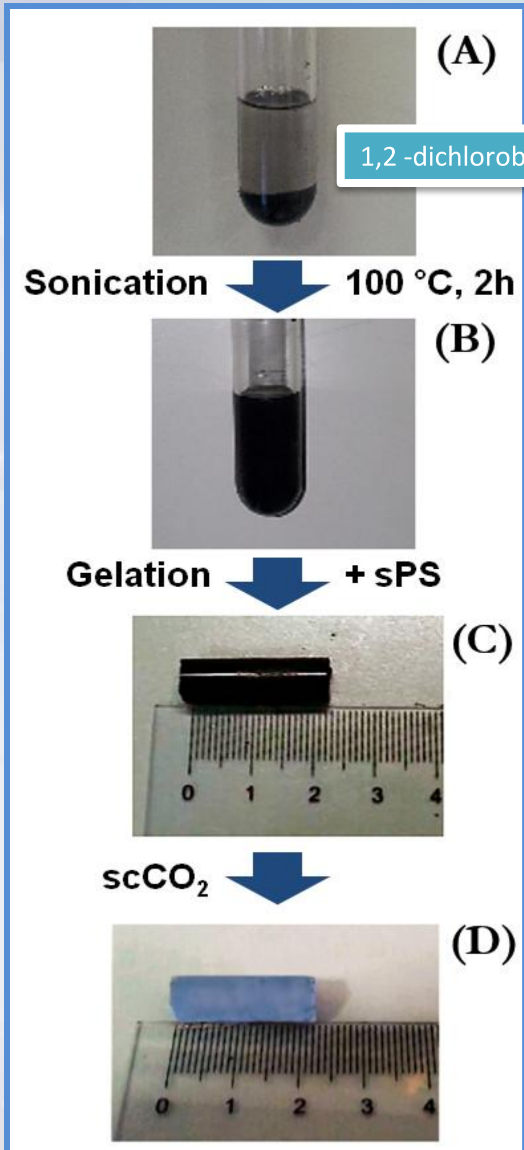
Hummers' method

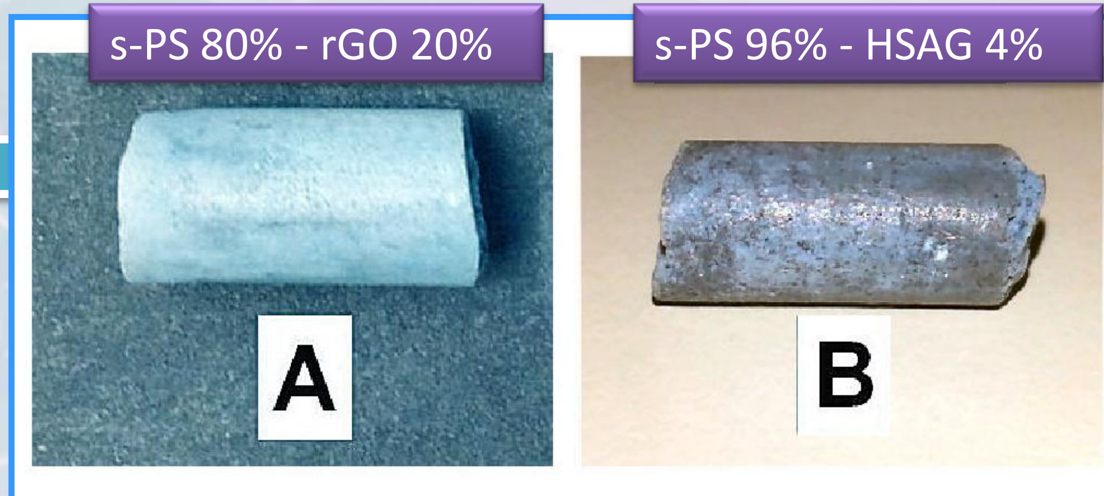
W.S. Hummers, R.E. Offeman, *J. Am. Chem. Soc.*, **1958** ,80,1339

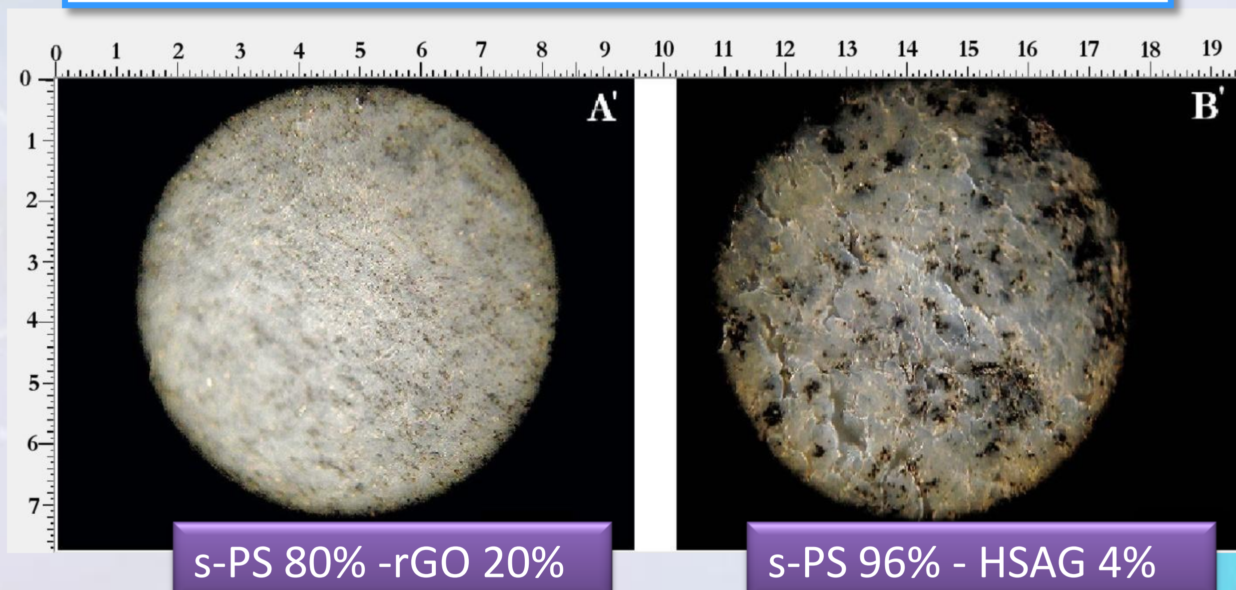
Starting Graphite



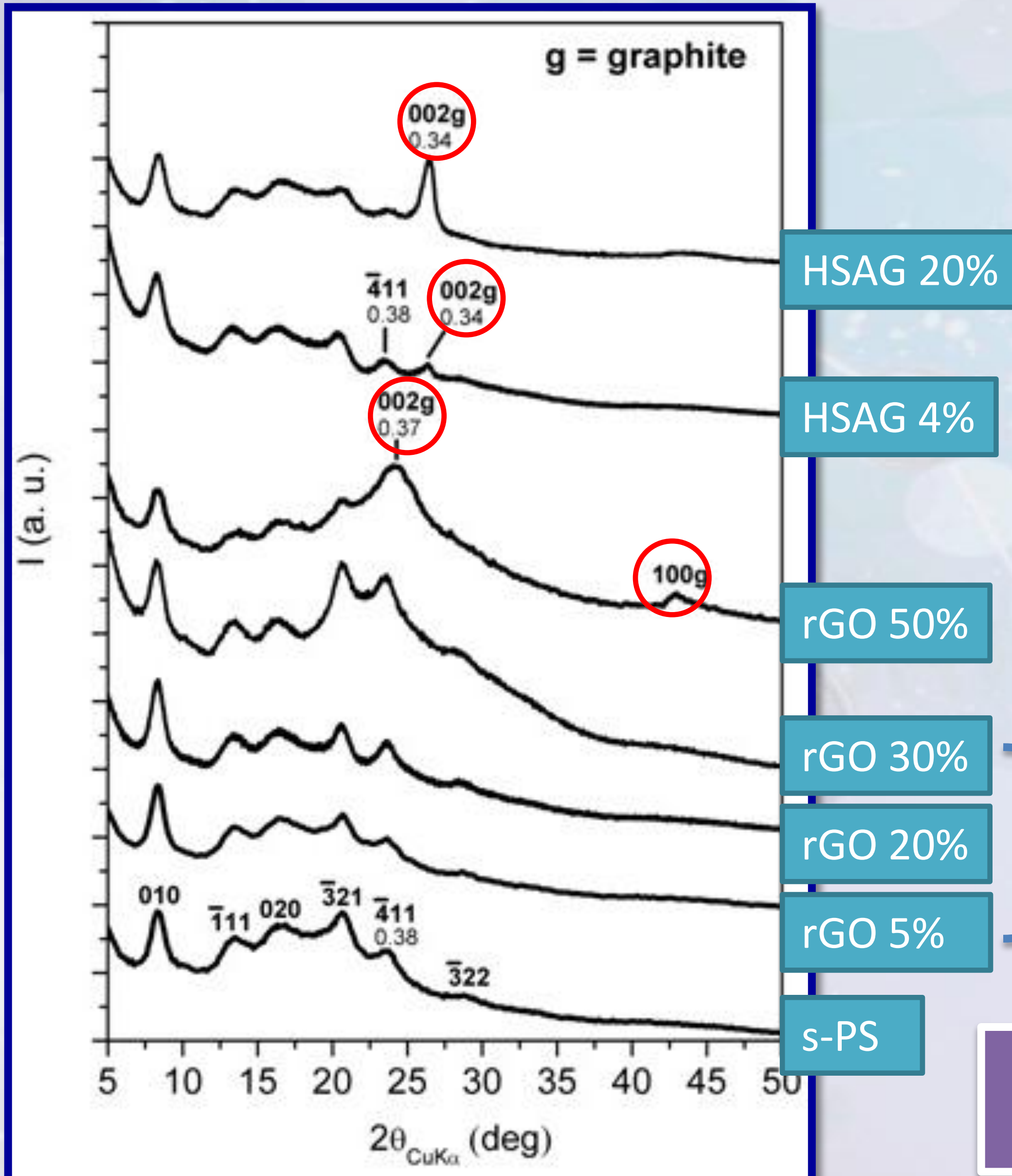
Nanocomposites Physically Crosslinked Polymeric Aerogels







Nanocomposites Physically Crosslinked Polymeric Aerogels



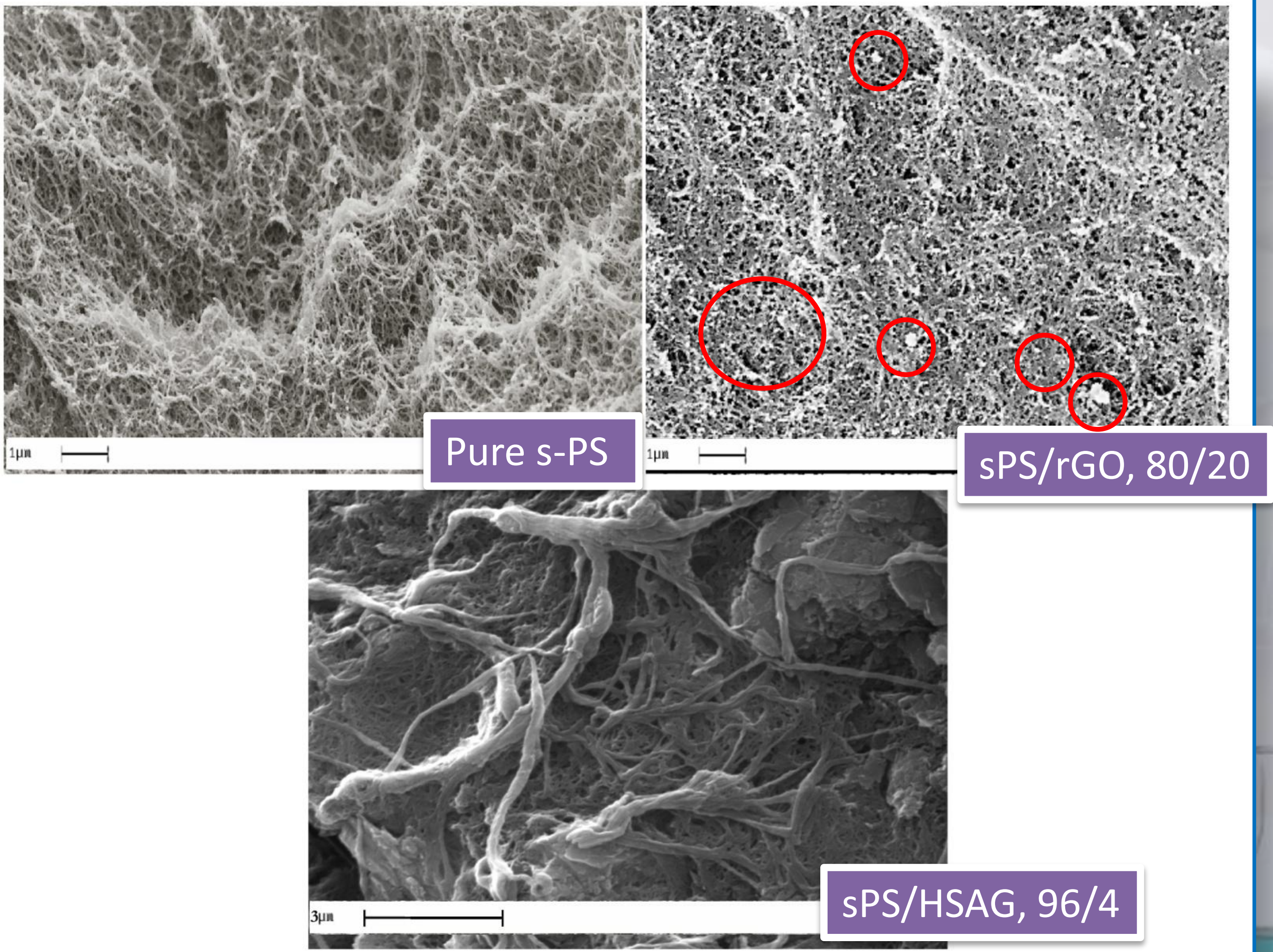
Absence of the narrow 002 peak of the rGO

↓

Negligible order in the direction perpendicular to the graphitic plane

Longo S., Mauro M., Daniel C., Musto P., Guerra G., *Submitted ACS Appl. Mater. Interfaces*

Nanocomposites Physically Crosslinked Polymeric Aerogels



Longo S., Mauro M., Daniel C., Musto P., Guerra G., *Submitted ACS Appl. Mater. Interfaces*

Nanocomposites Physically Crosslinked Polymeric Aerogels

BET measurements

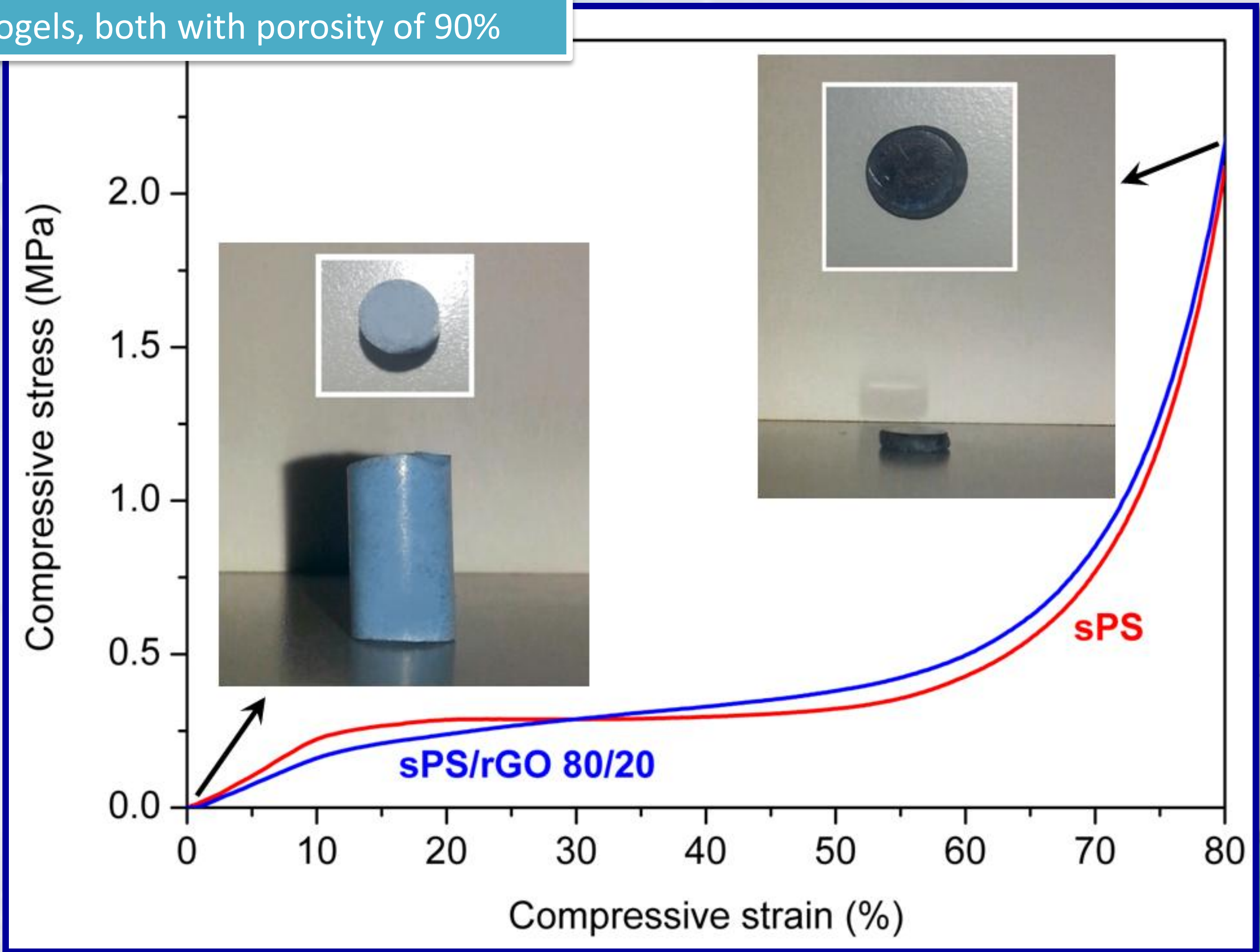
Sample	S_{BET}^{a} (m^2 g^{-1})
HSAG	308
sPS/HSAG, 80/20	**173**
GO	0.8
sPS/rGO 80/20 aerogel	**289**
sPS aerogel	312

Total area evaluated following the BET model in the standard $0.05<P/P_0<0.3$ pressure range.

Longo S., Mauro M., Daniel C., Musto P., Guerra G., *Submitted*

Nanocomposites Physically Crosslinked Polymeric Aerogels

Stress-strain curves in compression for aerogels, both with porosity of 90%



Longo S., Mauro M., Daniel C., Musto P., Guerra G., *Submitted*

Conclusions

1) Monolithic aerogels based on Polyethylene
- Thermal insulation
- Support for heterogeneous catalysis

2) Monolithic aerogels based on PPO
- Nanoporous crystalline phases of both PPO and sPS can be obtained for the 50/50 composition
- High and selective uptake of VOC

3) Monolithic aerogels based on PPPO
- Aerogels with high sorbent amorphous phase can be obtained for the PPPO 90 sPS 10 composition
- PPPO-sPS aerogels present higher uptake of VOC than commercial PPPO

4) Monolithic composite aerogels, with large amounts of OMMT and rGO, including the nanoporous-crystalline δ form of syndiotactic polystyrene (s-PS)
- Masterbatches for polymer composites
- Monolithic supported catalysts

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Prof. Diana Sannino

Dr. Felice de Santis
Dr. Stefano Cardea
Dr. Vincenzo Vaiano

Dr. Francesco di Renzo
Dr. Pellegrino Musto

Grazie!









Publications

•Daniel, C., **Longo, S**., Vitillo, J.G., Fasano, G., Guerra, G.(2011). *Chem. Mater.*23,3195-3200.

•Daniel, C., **Longo, S.**, Cardea, S., Vitillo, J.G., Guerra, G.(2012). *RSC Advances*, 2, 12011-12018.

•Daniel, C., **Longo, S**., Ricciardi, R., Reverchon, E., Guerra, G.(2013) *Macromol.Rapid Commun.*,*34*,1194-1207.

•**Longo ,S**., Vitillo J.G., Daniel C.,Guerra G. (2013).*ACS Appl. Mater. Interfaces* 5, 5493−5499.

•**Longo S.**, Mauro M., Daniel C., Galimberti M., Guerra G..(2013).*Frontiers in Polymer Chemistry*, 2013,1,1-9.

•**Longo S.**, Mauro M., Daniel C., Musto P., Guerra G., *Submitted*

•Daniel,C., **Longo, S.,** Galizia M. (2014). *Macromoluecular Symposia.*, 335, 70–77.

•Rizzo,P.,Ianniello,G.,**Longo,S.**,Guerra,G.(2013). *Macromolecules*. 46, 3995-4001.

•Vaiano V., Sacco O., **Longo S.**, Sannino D., Venditto V., Daniel C., Guerra G., Ciambelli P. (2013). SA2013A000011 Università di Salerno. Data deposito: 31/10/2013.

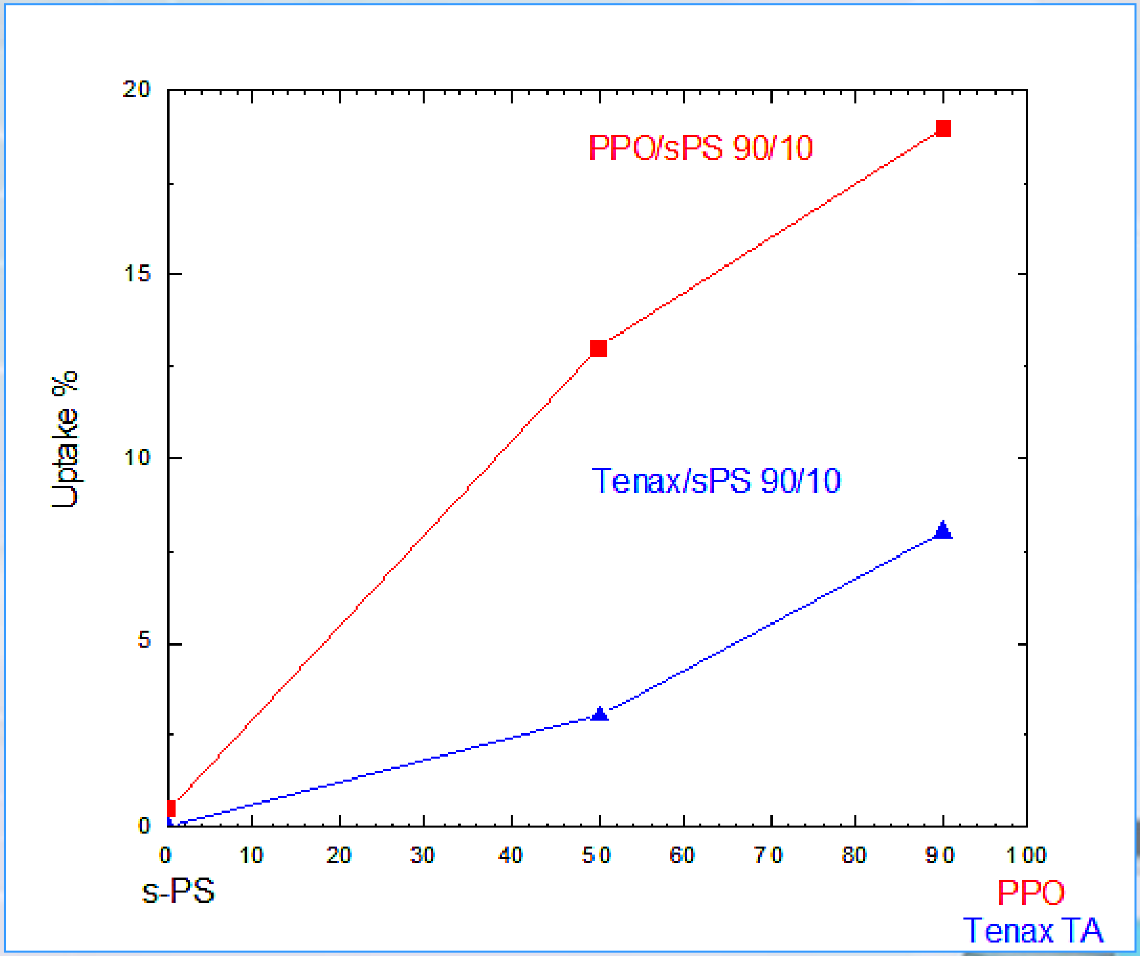
•Sannino, D., Vaiano, V.,Sacco, O.,Ciambelli, P.,**Longo S.**, Venditto, V., Guerra, G. "- *Submitted*

+ 8 Contributions at International Congresses

Thank you for your kind attention

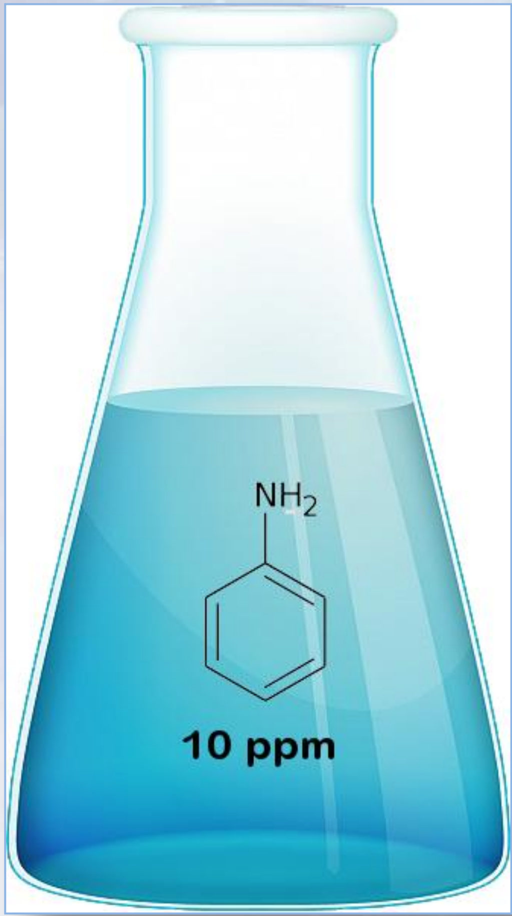
Second Year PhD Activity
Monolithic aerogels based on PPPO:sorption properties

3 days of sorption



S.Longo, J.G.Vitillo, C.Daniel, G.Guerra, *ACS Applied Materials & Interfaces*, **2013**

Sorption from diluted aqueos solutions – aniline 10ppm



+

PPO/sPS 50-50

→

3 days of sorption

Aniline uptake

8 wt%

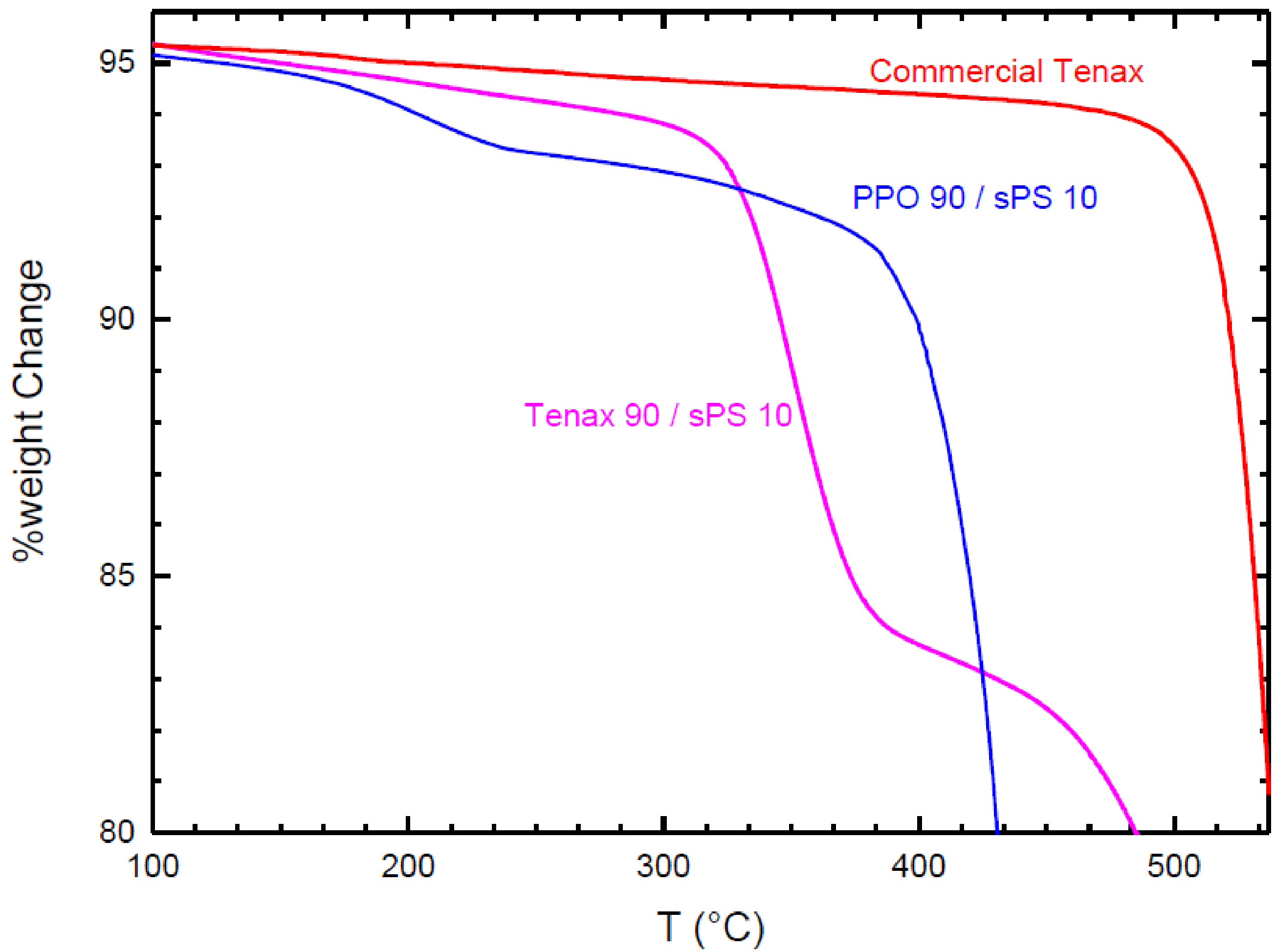
Properties of aerogels based on nanoporous-crystalline PPO

x_{PPO}	C_{pol} (wt%)	$x_{PPO,crys}$	S_{BET}[a] ($m^2\ g^{-1}$)	S_{micro}[b] ($m^2\ g^{-1}$)	χ_{PPO} (%)
s-PS	**20**	**0**	**206**	**27**	**0**
0.25	20	0	217	49	0
0.5	20	0.24	337	118	48
0.75	20	0.27	341	141	36
0.9	20	0.53	483	172	58
PPO (powder)	**25**	**0.59**	**535**	**195**	**59**

[a]Total area evaluated following the BET model in the standard 0.05 < P/P_0 < 0.25 pressure range. [b]Micropore area obtained from the t-plot

Monolithic aeogels based on Tenax TA :sorption properties

Pyrene uptake



Intrinsic Mass Solubility = 4.4 x 10 $^{-5}$ g/L

Monolithic physically crosslinked polymeric aerogels

The importance of obtaining an aerogel

Large surface aeras

Faster adsorption kinetics

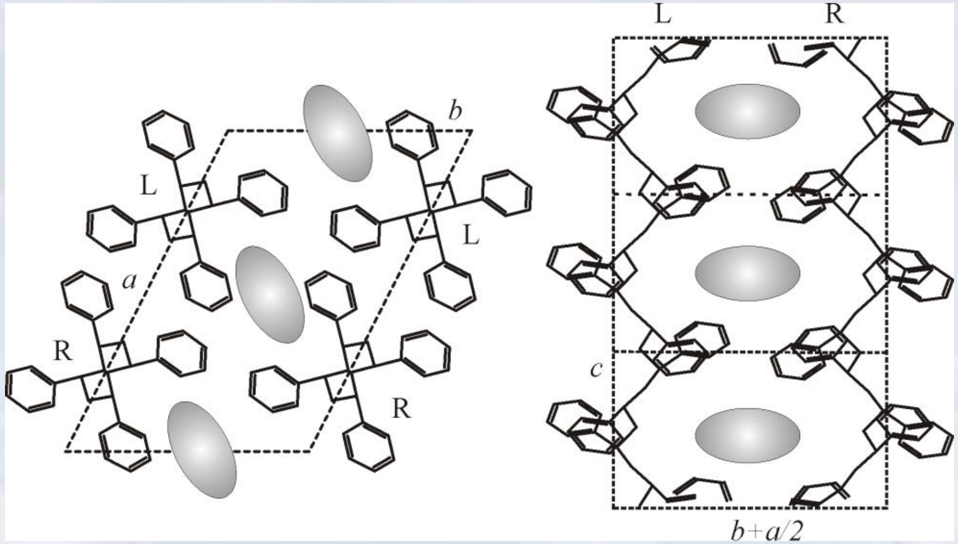
Fine Dust Hazard

Nanoporous Crystalline Phases of s-PS

Cavities

δ

cavities volume ≈ 120 Å^3



Top view

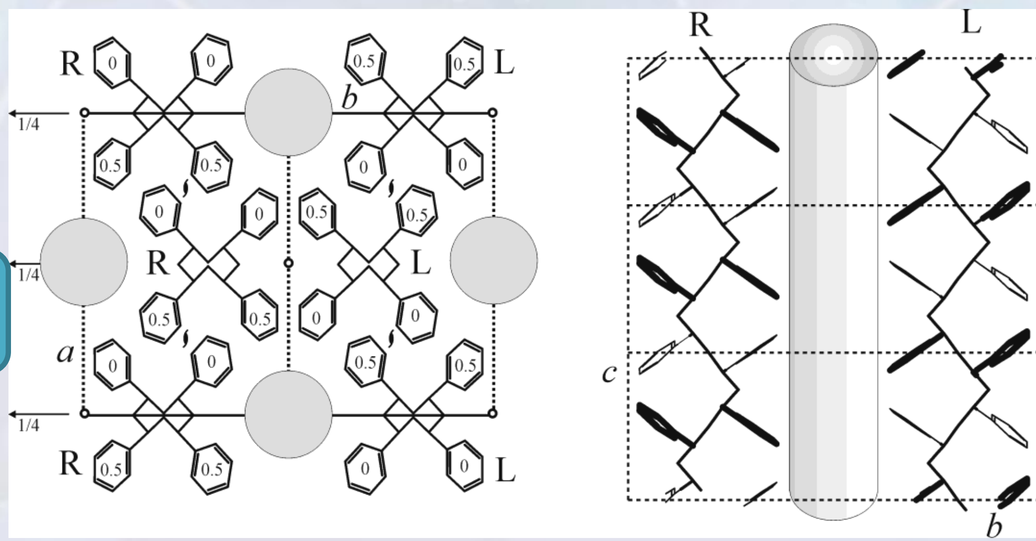
Lateral view

De Rosa, Guerra, Petraccone, Pirozzi *Macromolecules* **1997**, *30,* 4147

Channels

ε

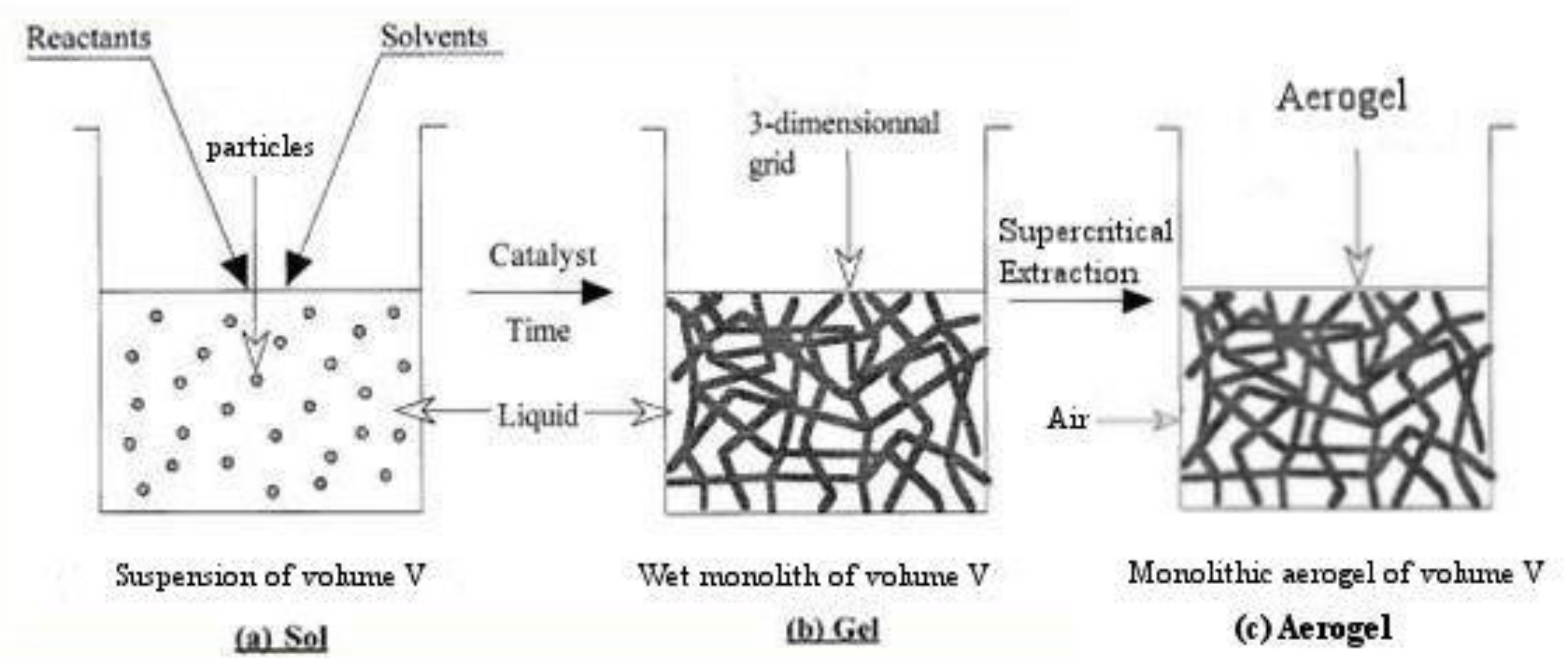
channel diameter ≈ 5 Å



Petraccone, Ruiz, Tarallo, Rizzo, Guerra, *Chem.Mater.* **2008**, *20,* 3663

Monolithic crosslinked aerogels

Sol-Gel process



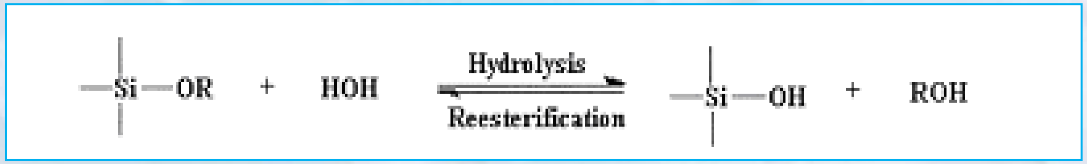
S.S.,Kistler,*Nature* **1931**,127,141.
A.C.,*Pierre;*G.M.Pajonk.*Chem Rev*.**2002**,102,4243-4265.

Monolithic crosslinked aerogels

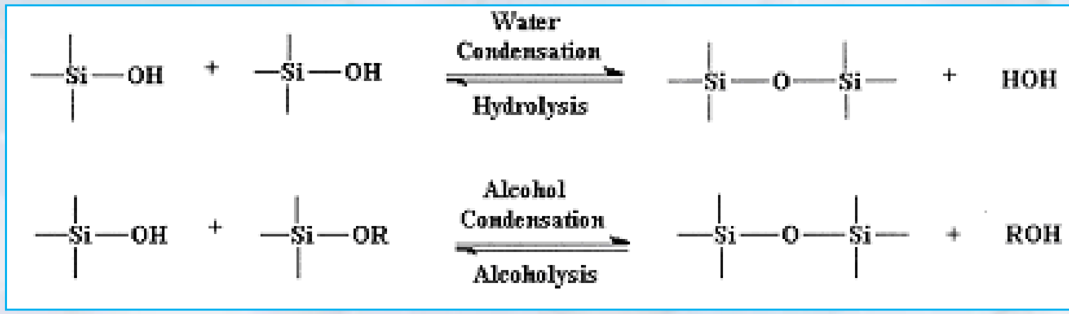
Inorganic Aerogels

Silicon Precursor: $Si(OR)_4$ where R is often an alkyl $-CH_3$

Hydrolysis

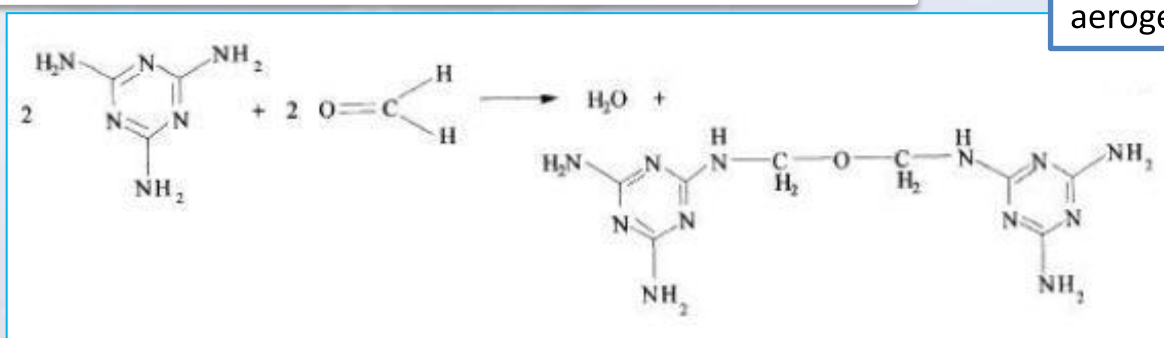


Condensation



Polymeric Aerogels Chemically Crosslinked

Melamine-formaldehyde aerogels



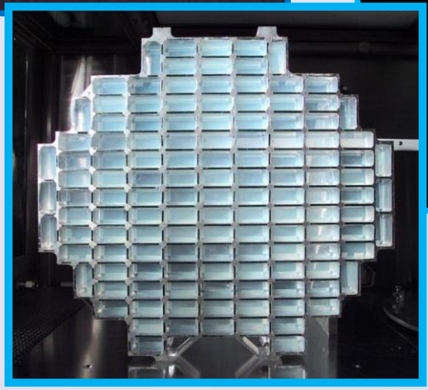
A.C.,*Pierre;*G.M.Pajonk.*Chem Rev*.**2002**,102,4243-4265

Monolithic inorganic aerogels

Aerogel consisting of inorganic oxides

Silica aerogel is the most common type of aerogel and the most extensively studied and used

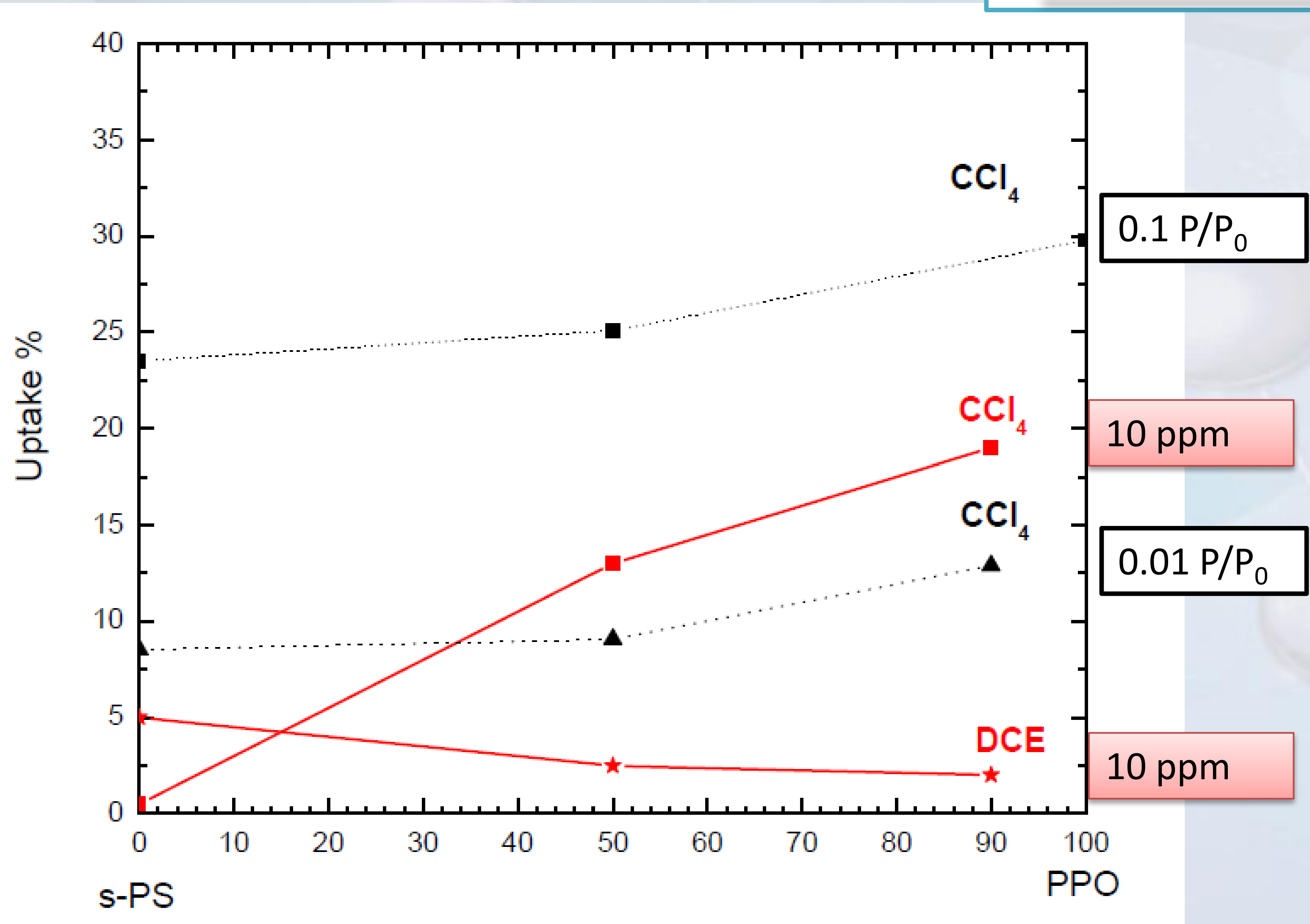
- ✓Silica
- ✓Alumina
- ✓Zirconia
- ✓Stannic or tungsten oxide



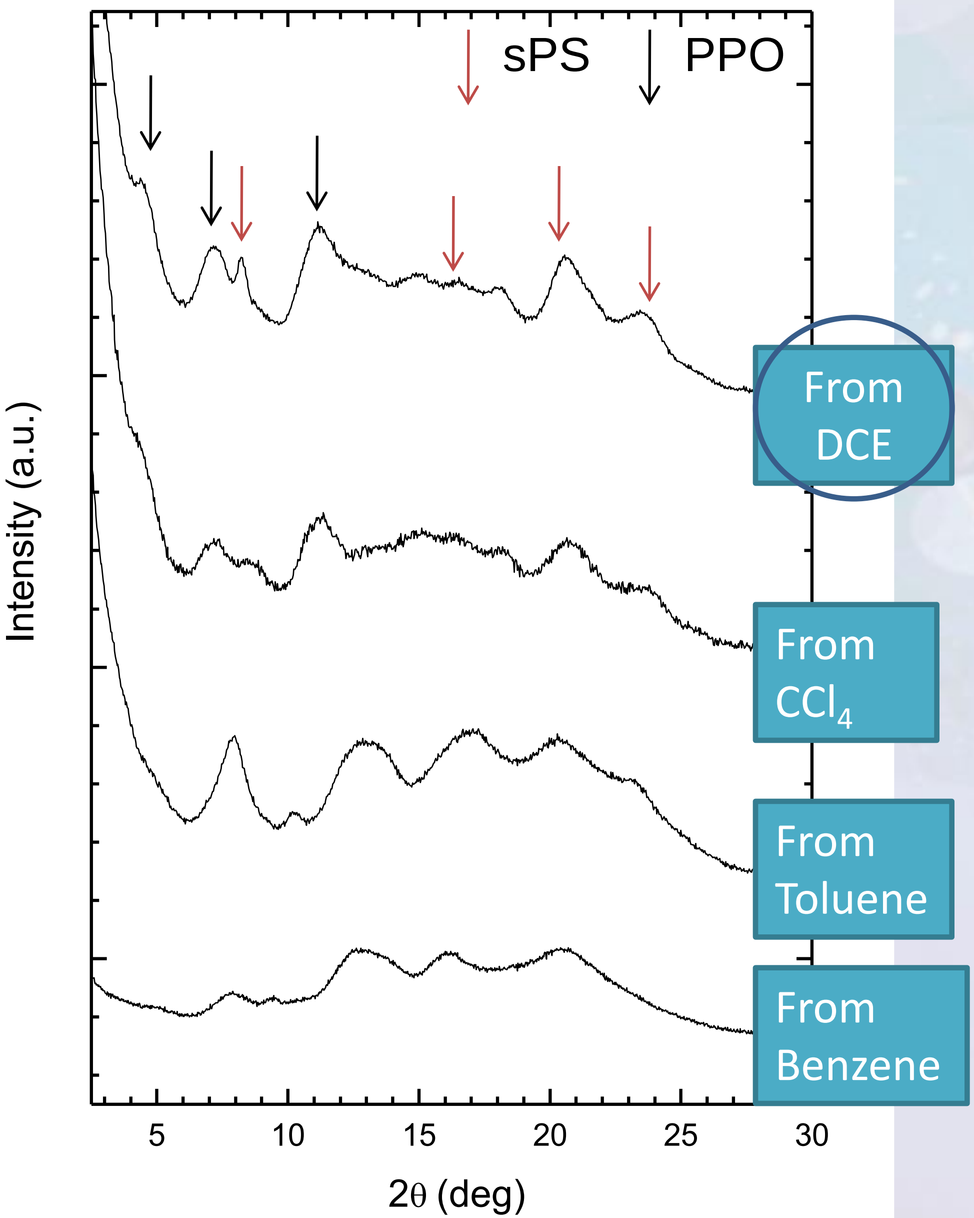
H.D,Gesser; P.C.,Goswami;*Chem.Rev* **1989**,89,765-788

Monolithic Nanoporous Crystalline Aerogels based on PPO

Typical crystalline pores of both PPO and s-PS



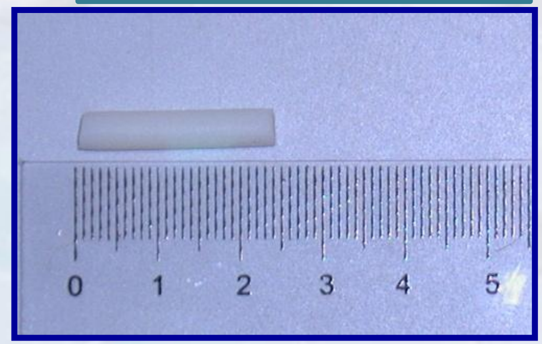
First Year PhD Activity:
Monolithic Nanoporous Crystalline Aerogels based on PPO



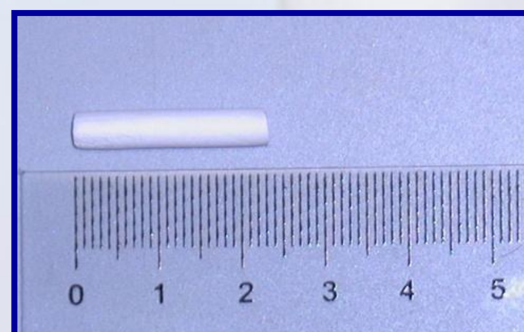
$\chi_{PPO}=0.50$

$\chi_{sPS}= 0.50$

Porosity 80%



CO_2 Extraction



First Year PhD Activity: Sorption Properties
Sorption from diluted aqueos solutions –CCl_4 10ppm

3 days of sorption

+

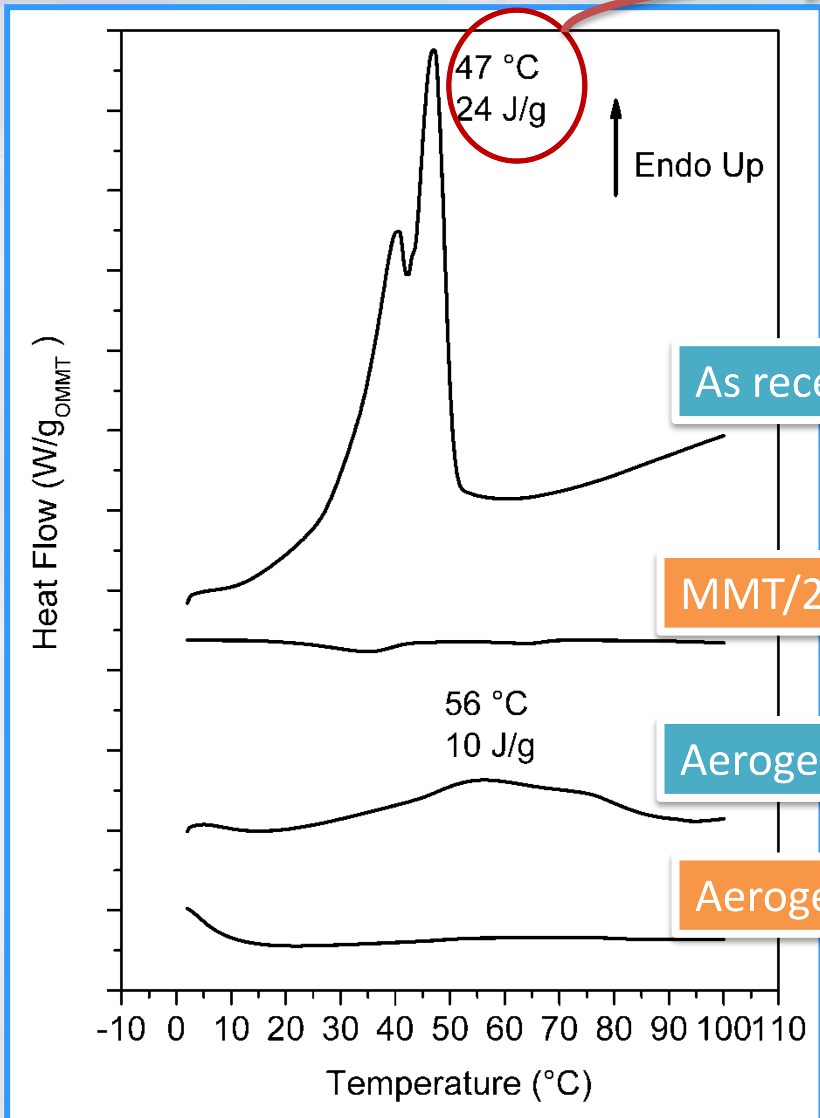
CCl_4 uptake

1. Aerogel δ sPS → 0.5wt%
2. Aerogel PPO/sPS 50-50 → 13 wt%
3. Aerogel PPO/sPS 90-10 → 19 wt%

Third Year PhD Activity: Nanocomposites Physically Crosslinked Polymeric Aerogels

OMMT exfoliation by $scCO_2$

loss of rotator order of the hydrocarbon tails of the cations intercalated in the interlayer space



As received MMT/2HT

MMT/2HT after treatment by $scCO_2$

Aerogels 50/50 s-PS/as received OMMT

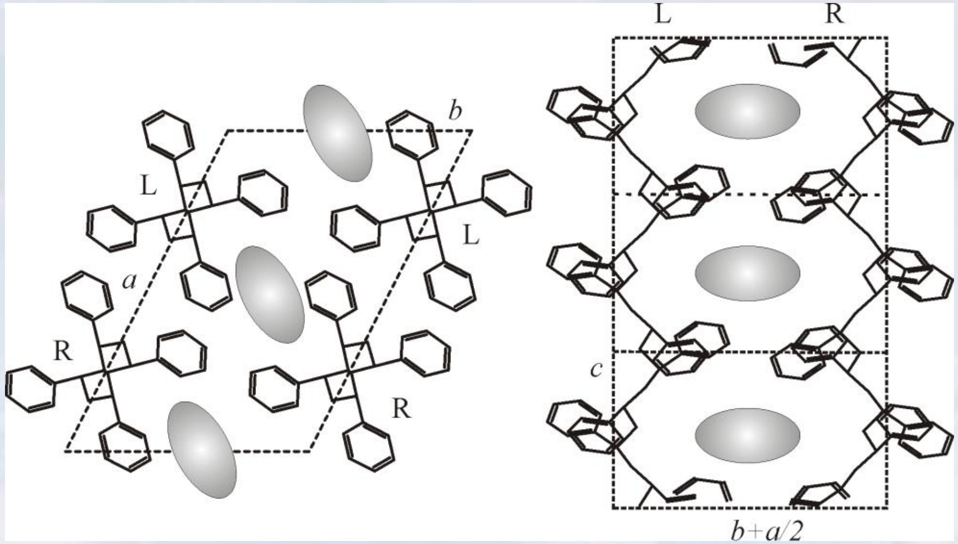
Aerogels 50/50 s-PS/exfoliated OMMT

Nanoporous Crystalline Phases of s-PS

Cavities

δ

cavities volume ≈ 120 Å^3



Top view

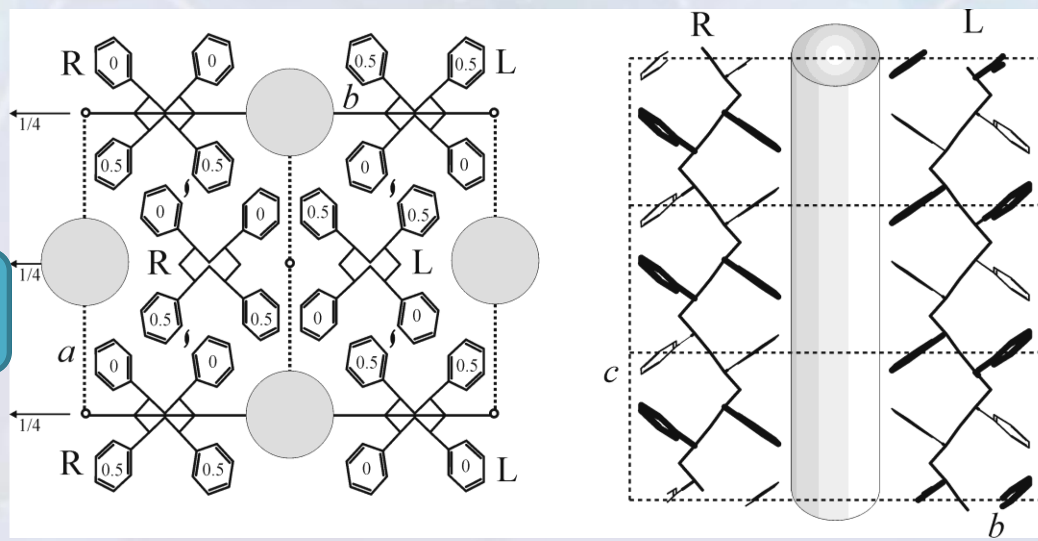
Lateral view

De Rosa, Guerra, Petraccone, Pirozzi *Macromolecules* **1997**, *30,* 4147

Channels

ε

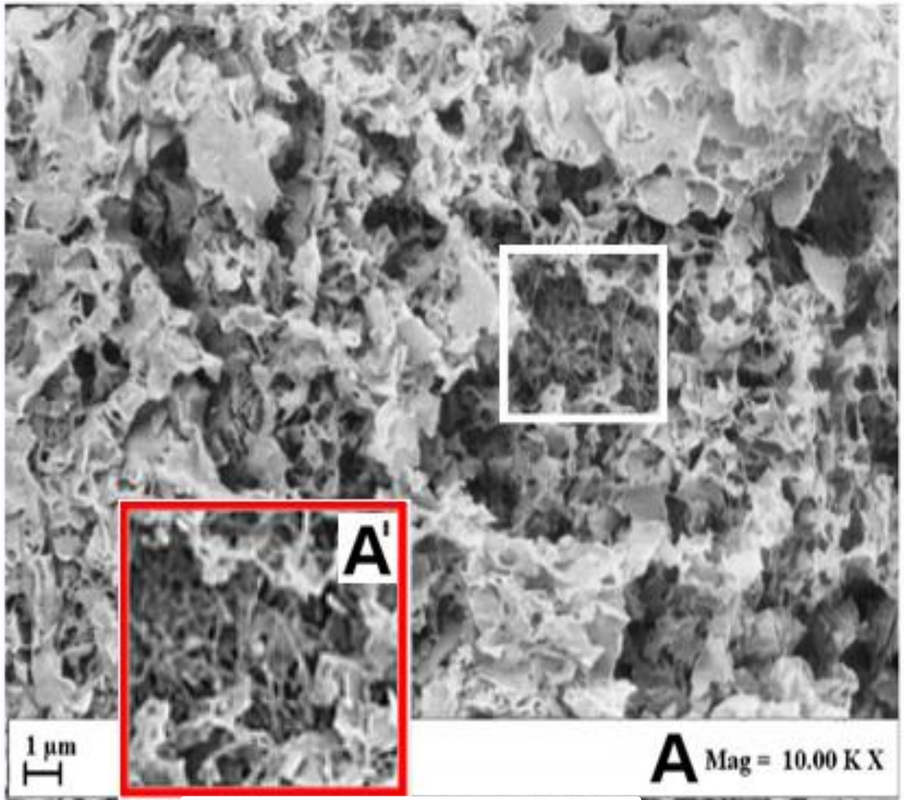
channel diameter ≈ 5 Å



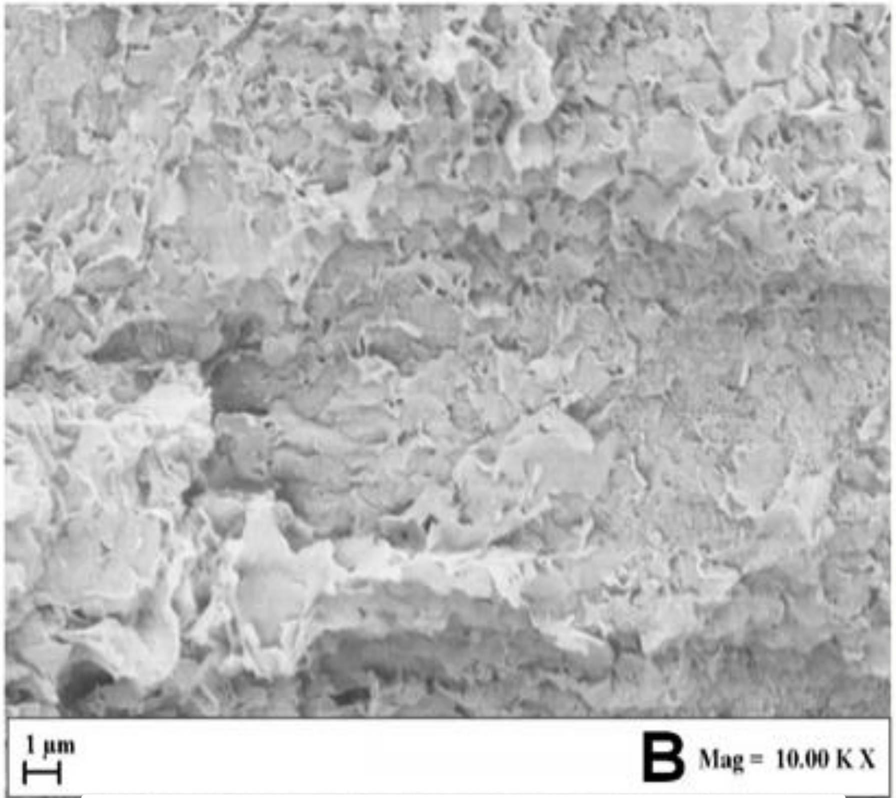
Petraccone, Ruiz, Tarallo, Rizzo, Guerra, *Chem.Mater.* **2008**, *20,* 3663

Third Year PhD Activity: Nanocomposites Physically Crosslinked Polymeric Aerogels

80/20 weight ratio
P=90%



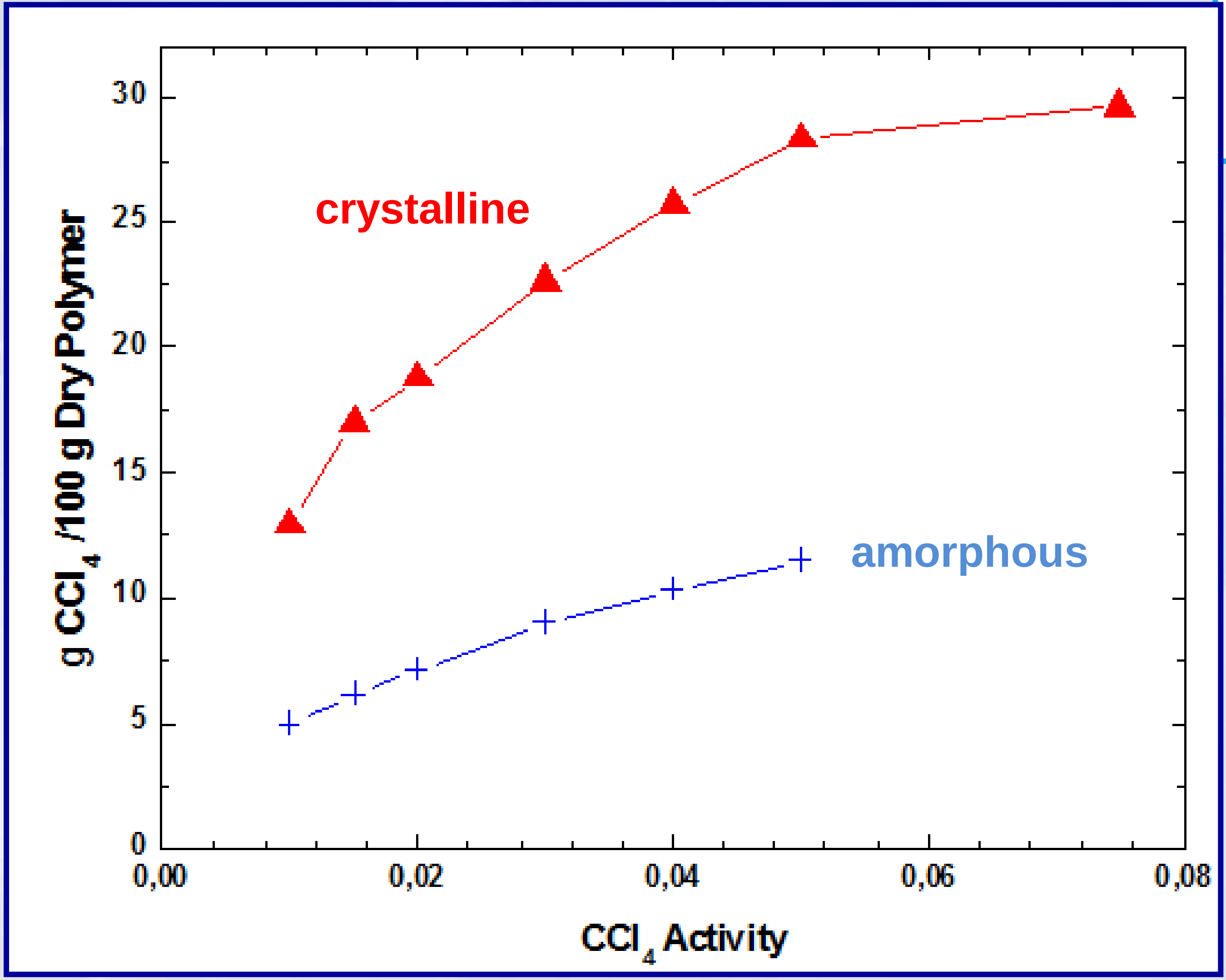
s-PS aerogels with exfoliated OMMT

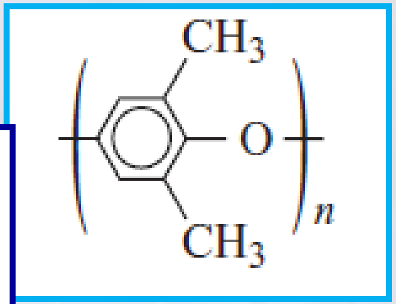


s-PS aerogels with as received OMMT

Polymeric aerogels with nanoporous crystalline phase

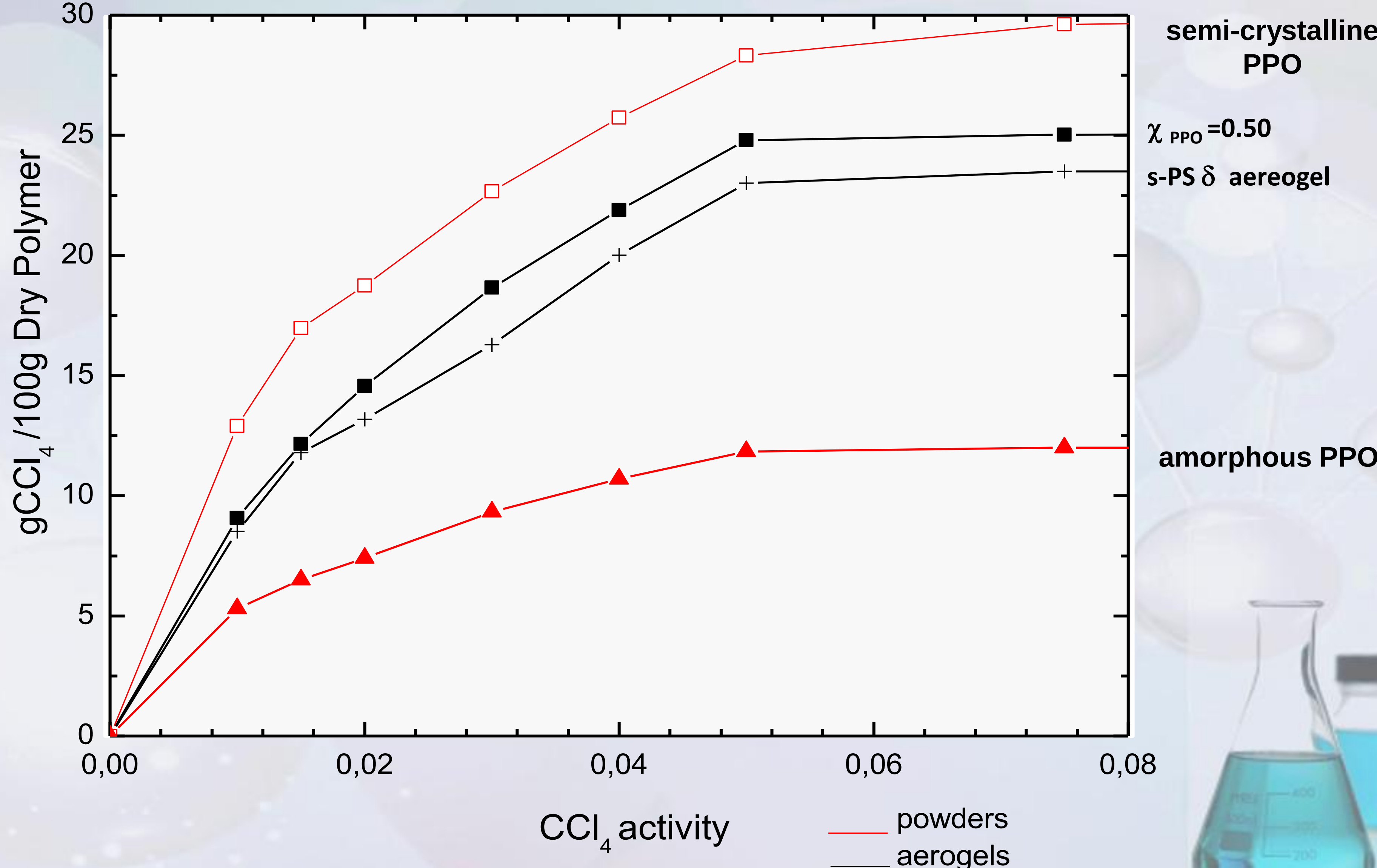
Poly(2,6-dimethyl-1,4-phenylenoxide) (PPO)





Daniel, C.; Longo, S.;Vitillo, J.G.;Fasano, G.;Guerra, G. *Chem. Mater*. **2011**, *23*, 3195

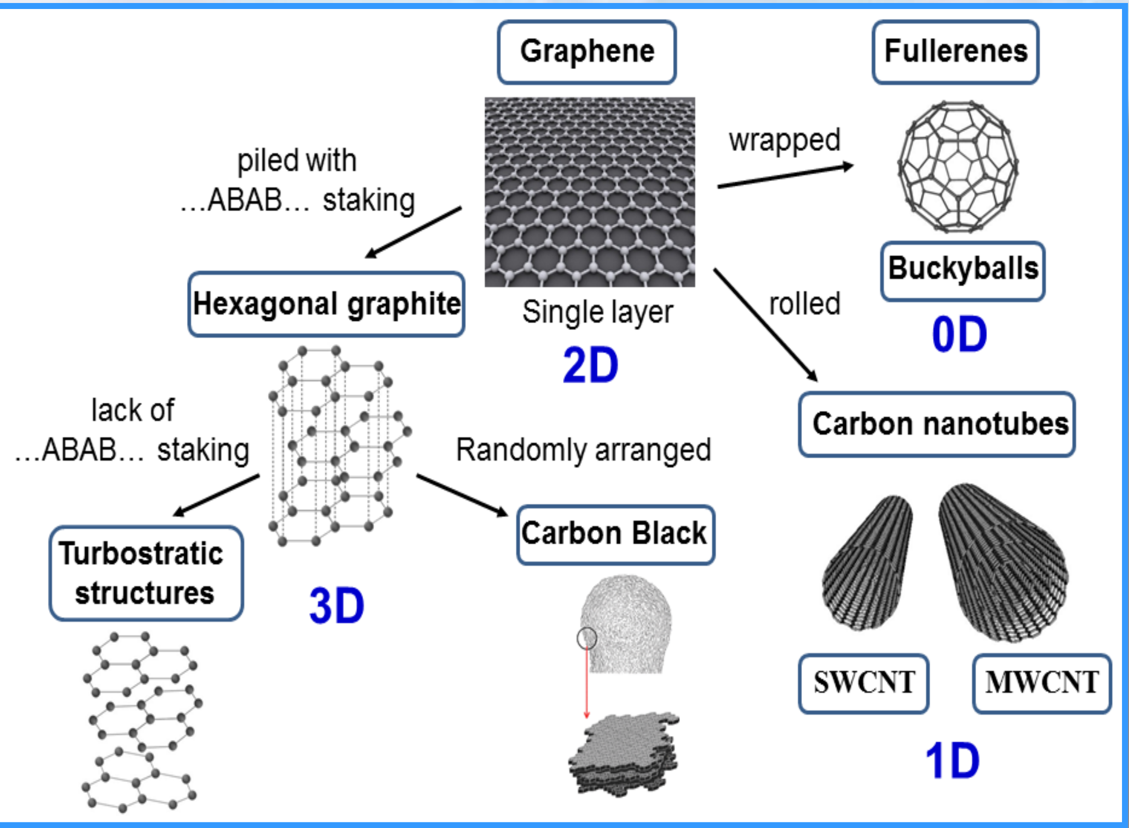
First Year PhD Activity: Sorption Properties
Volatile organic compounds (VOCs) from vapors at low activity



Third Year PhD Activity: Nanocomposites Physically Crosslinked Polymeric Aerogels

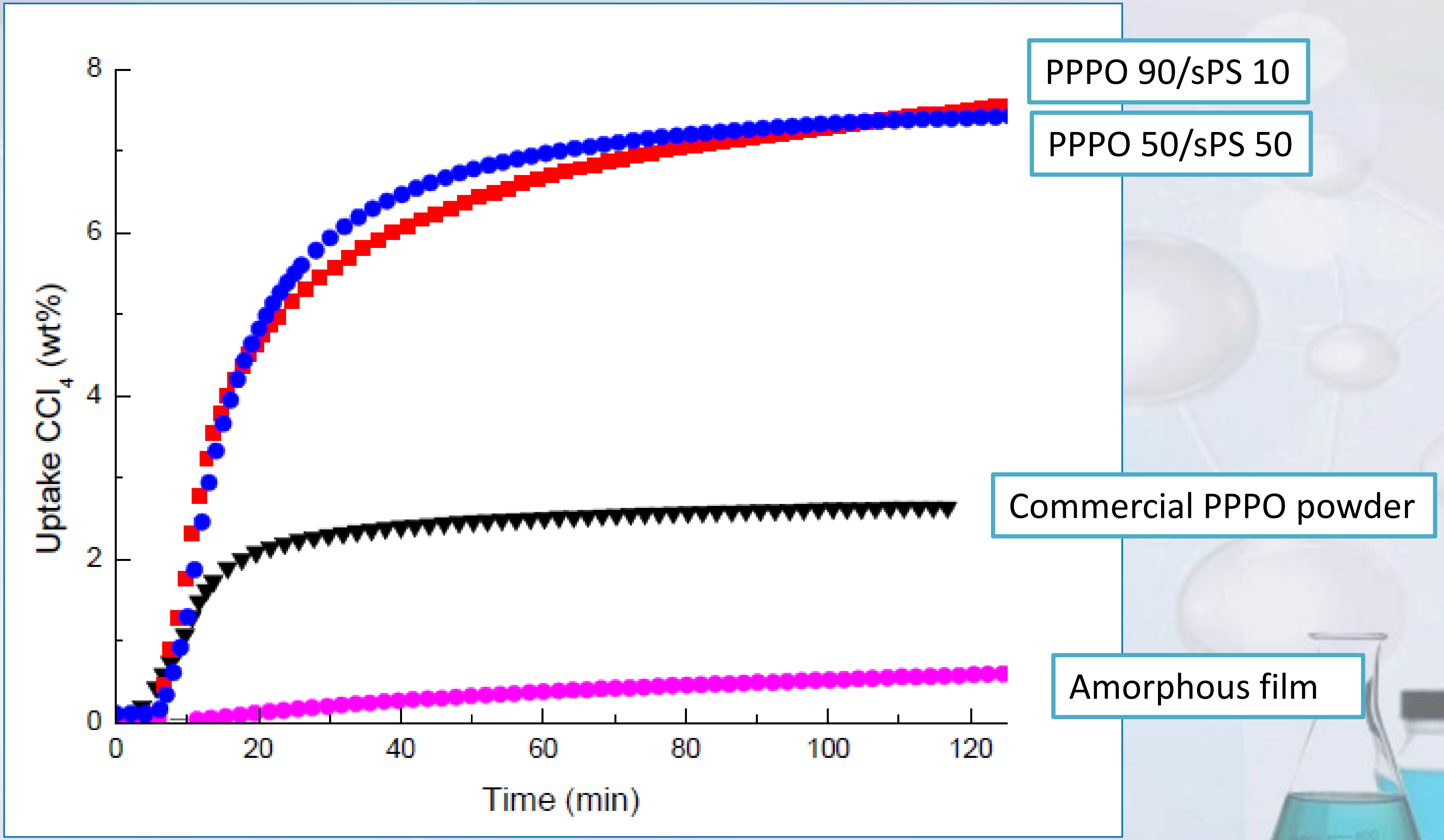
Graphene Preparation

- Micromechanical exfoliation
- Epitaxial growth on silicon carbide
- Chemical vapor deposition (CVD)
- Arc discharge method
- Small molecules intercalation within multilayered graphite
- Unzipping of CNTs
- Electrochemical method
- **Chemical reduction of exfoliated GO**



a) P. Avouris, F. Xia, *MRS Bull.*, **2012**, 37,1225. b) N. Zhang, Y. Zhang, Y.J. Xu, *Nanoscale,* **2012** ,4, 5792. c) T.H. Tsai, S.C. Chiou, S.M. Chen, *Int. J. Electrochem. Sci.*, **2011**, 6 ,3333. d) K. Zhang, L.L. Zhang, X.S. Zhao, J. Wu, *Chem. Mater.* **2010**, 22,1392. e) K. Yang, L. Feng, X. Shi, Z. Liu, *Chem. Soc. Rev.*, **2013**, 42, 530. f) V. Mani, A.P. Periasamy, S.M. Chen, *Electrochem. Commun.* ,**2012**, 1775 g) S.W. Ting, A.P. Periasamy, S.M. Chen, R. Saraswathi, *Int. J. Electrochem. Sci.*,**2011** ,6,4438.

Second Year PhD Activity
Monolithic aerogels based on PPPO:sorption properties



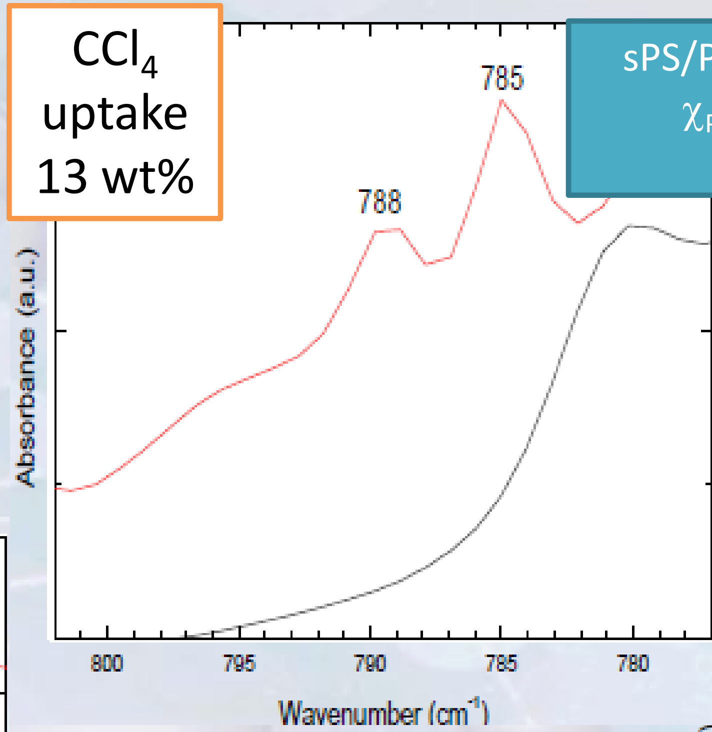
First Year PhD Activity: Sorption Properties
Sorption from diluted aqueous solutions – CCl_4 10ppm

CCl_4 10ppm

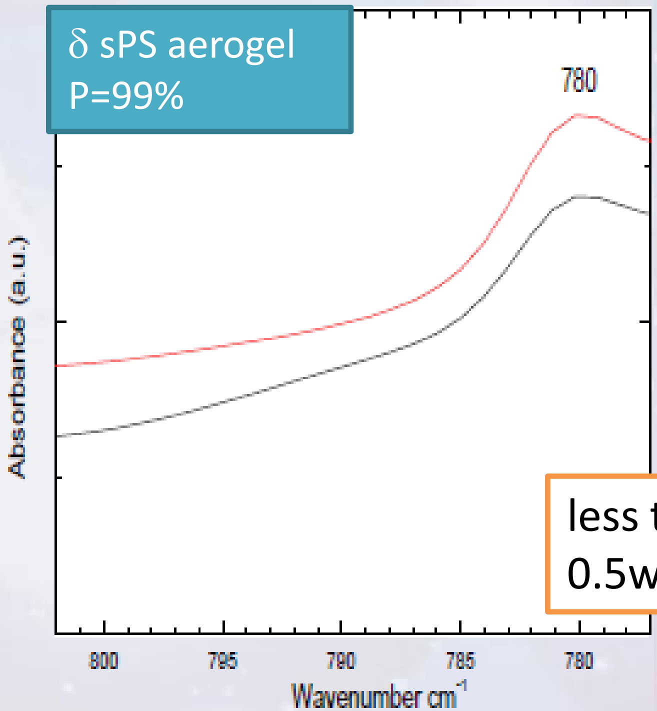
3 days of sorption

CCl_4 uptake 13 wt%

sPS/PPO aerogel
χ_{PPO}=0,50
P=80%

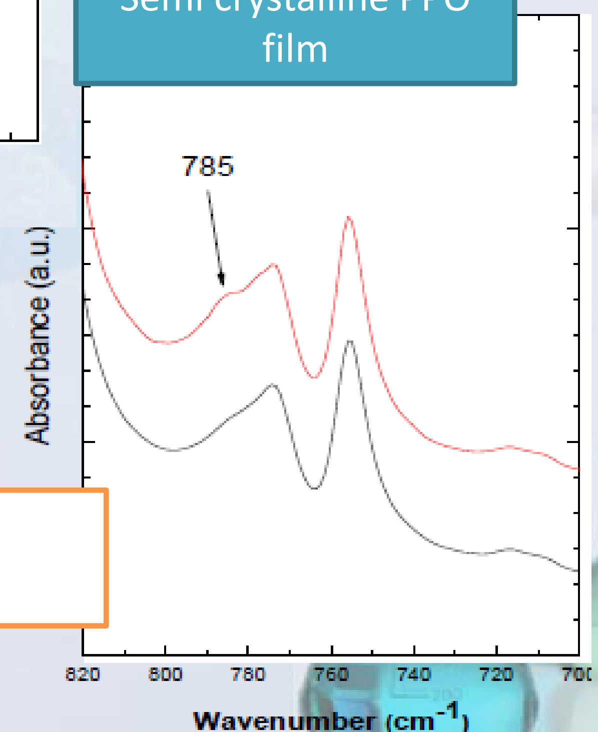


δ sPS aerogel
P=99%



less than 0.5wt%

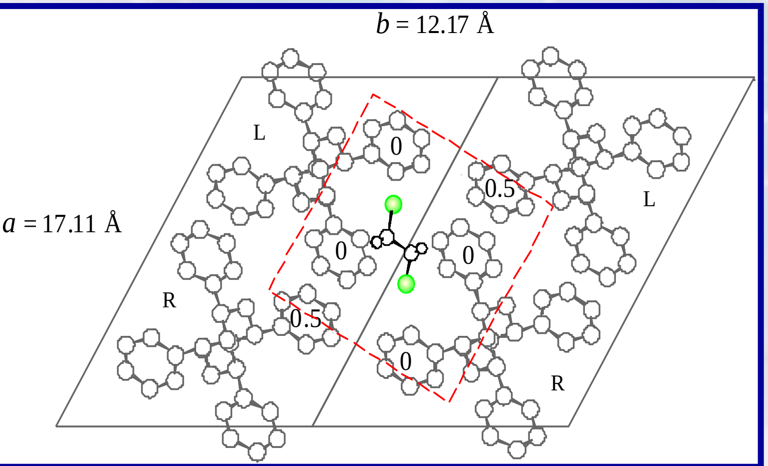
Semi crystalline PPO film



less than 1wt%

First Year PhD Activity: Sorption Properties
Sorption from diluted aqueous solutions –DCE 10ppm

Only trans conformer is included into δ sPS crystalline phase



Daniel, C.; Sannino, D.; Guerra, G. *Chem.Mater.* **2008**, *20*,577-582

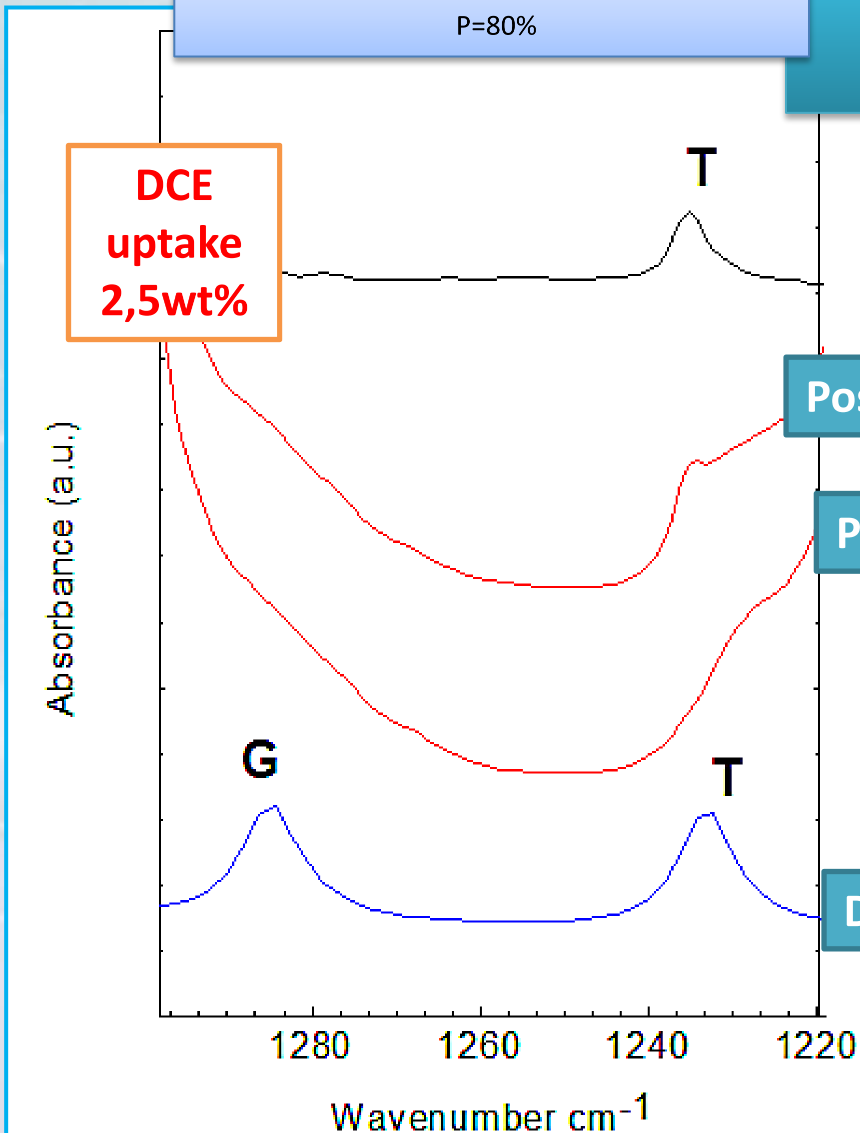
δsPS aerogel
P=99%

→ 5wt%

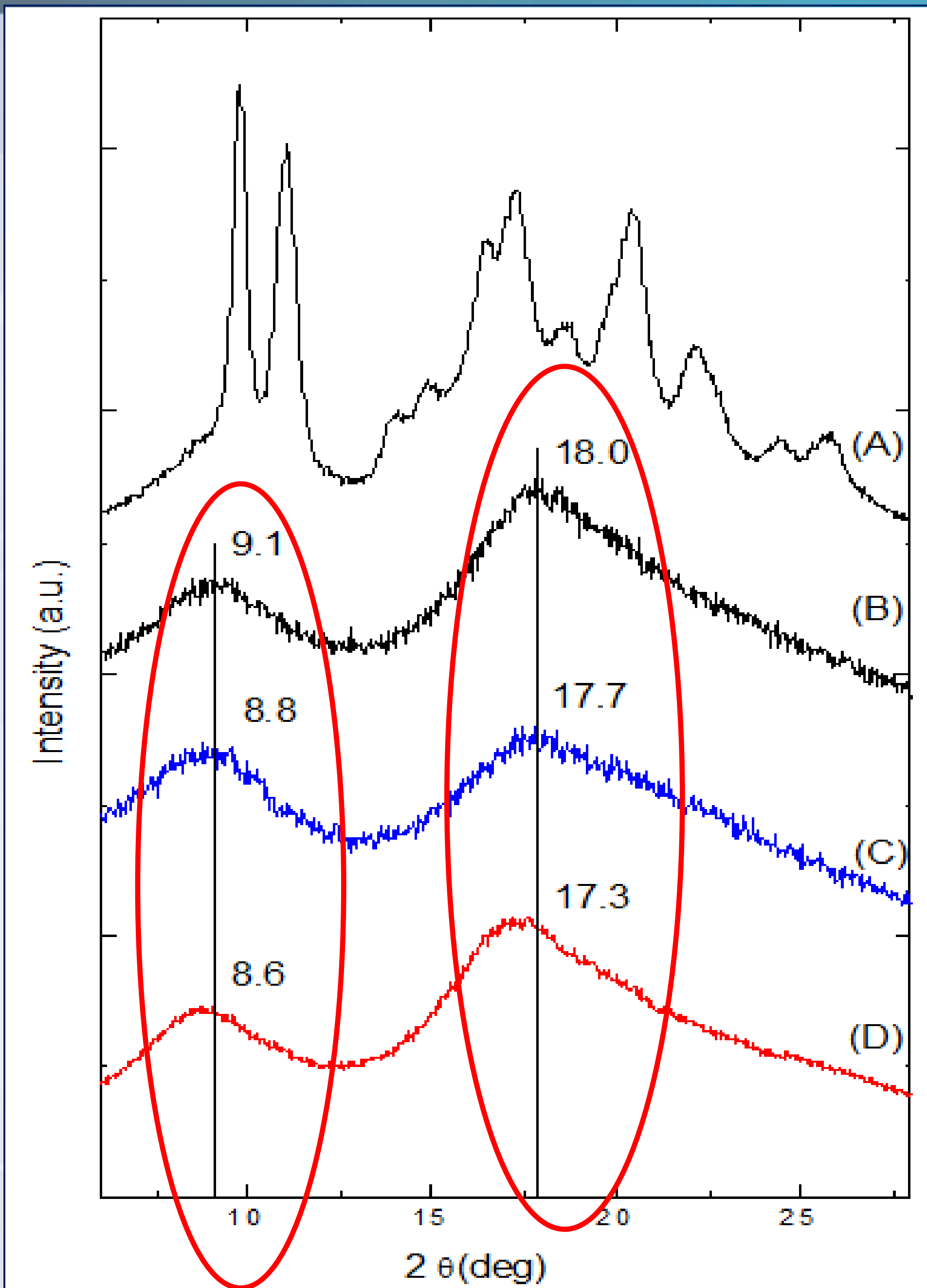
sPS/PPO aerogel χ_{PPO}=0,50
P=80%

1 days sorption

DCE uptake 2,5wt%



PPPO properties



S.Longo, J.G.Vitillo, C.Daniel, G.Guerra, *ACS Applied Materials & Interfaces*, **2013**

Commercial PPPO powder

Amorphous films obtained by casting in $CHCl_3$ at 20°C

d_{max} = 4.9 Å

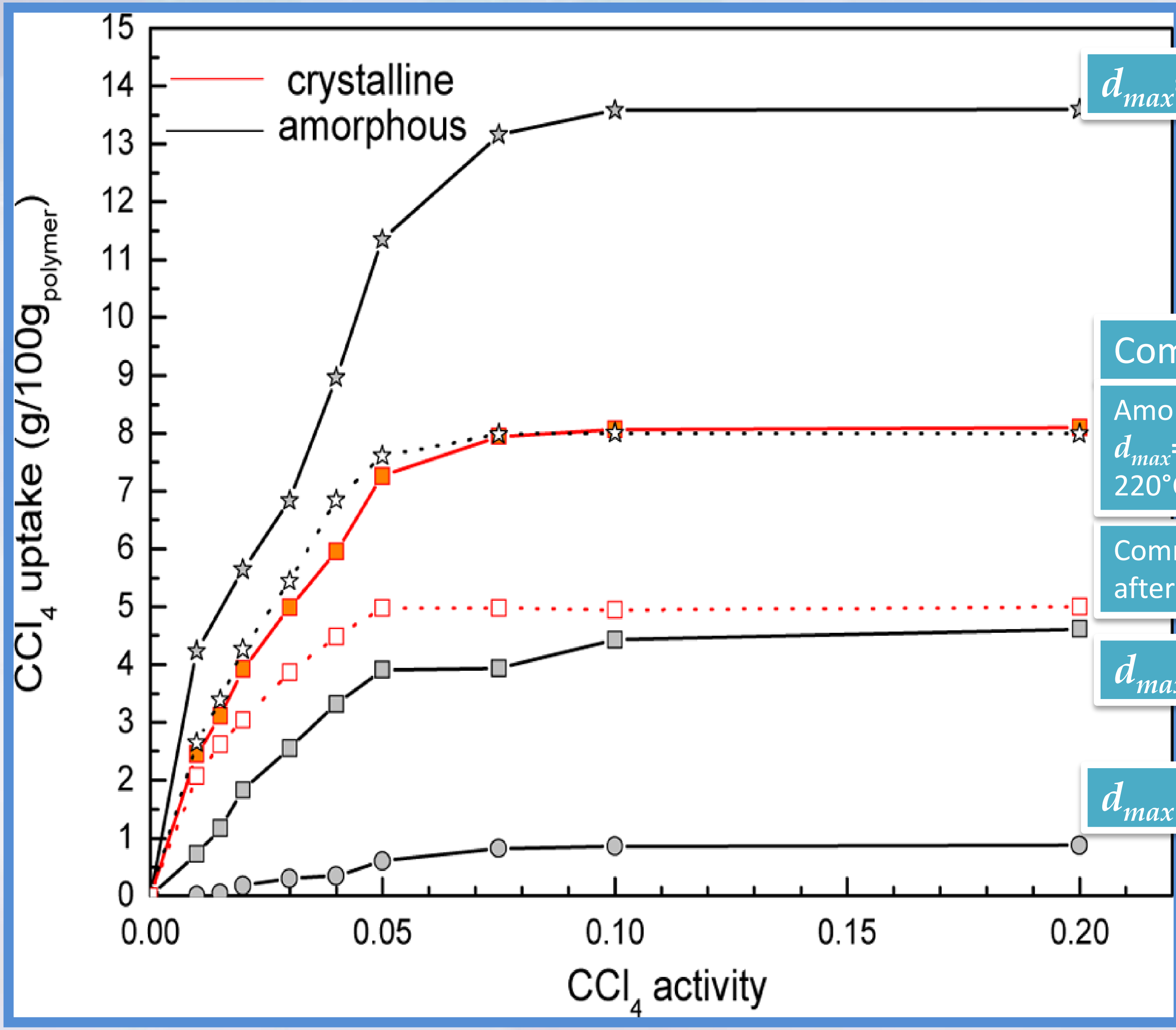
Amorphous films obtained by casting in $CHCl_3$ at 135°C

d_{max} = 5.00 Å

Amorphous powder obtained by concentrated solution (C_{pol}=20%) , successively extracted by supercritical CO_2 .

d_{max}= 5.12 Å

PPPO properties



d_{max}= 5.12 Å

Commercial PPPO

Amorphous powder PPPO
d_{max}= 5.12 Å after annealing at 220°C

Commercial PPPO
after annealing at 220°C

d_{max} = 5.00 Å

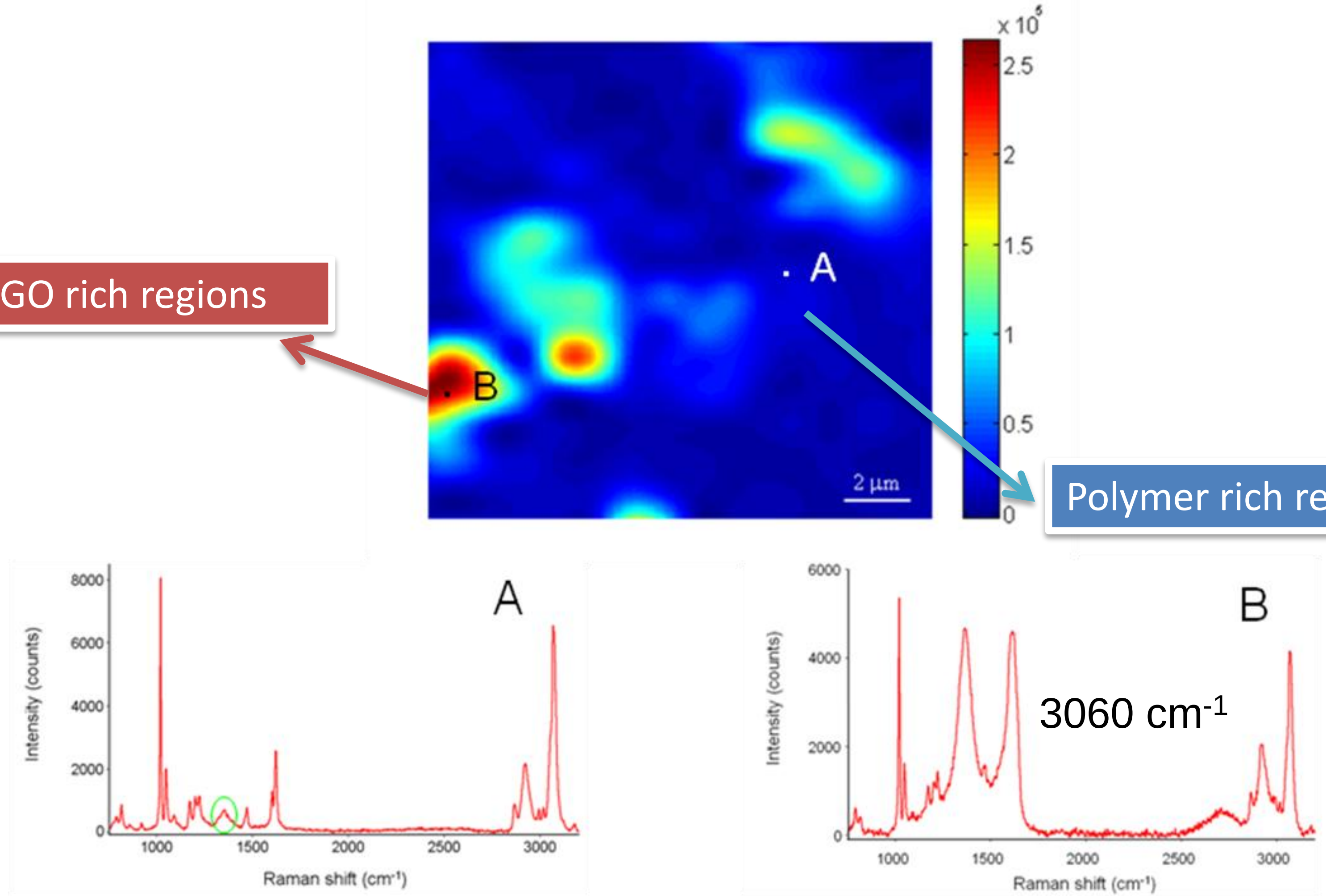
d_{max} = 4.9 Å

S.Longo, J.G.Vitillo, C.Daniel, G.Guerra, *ACS Applied Materials & Interfaces*, **2013**

Nanocomposites Physically Crosslinked Polymeric Aerogels

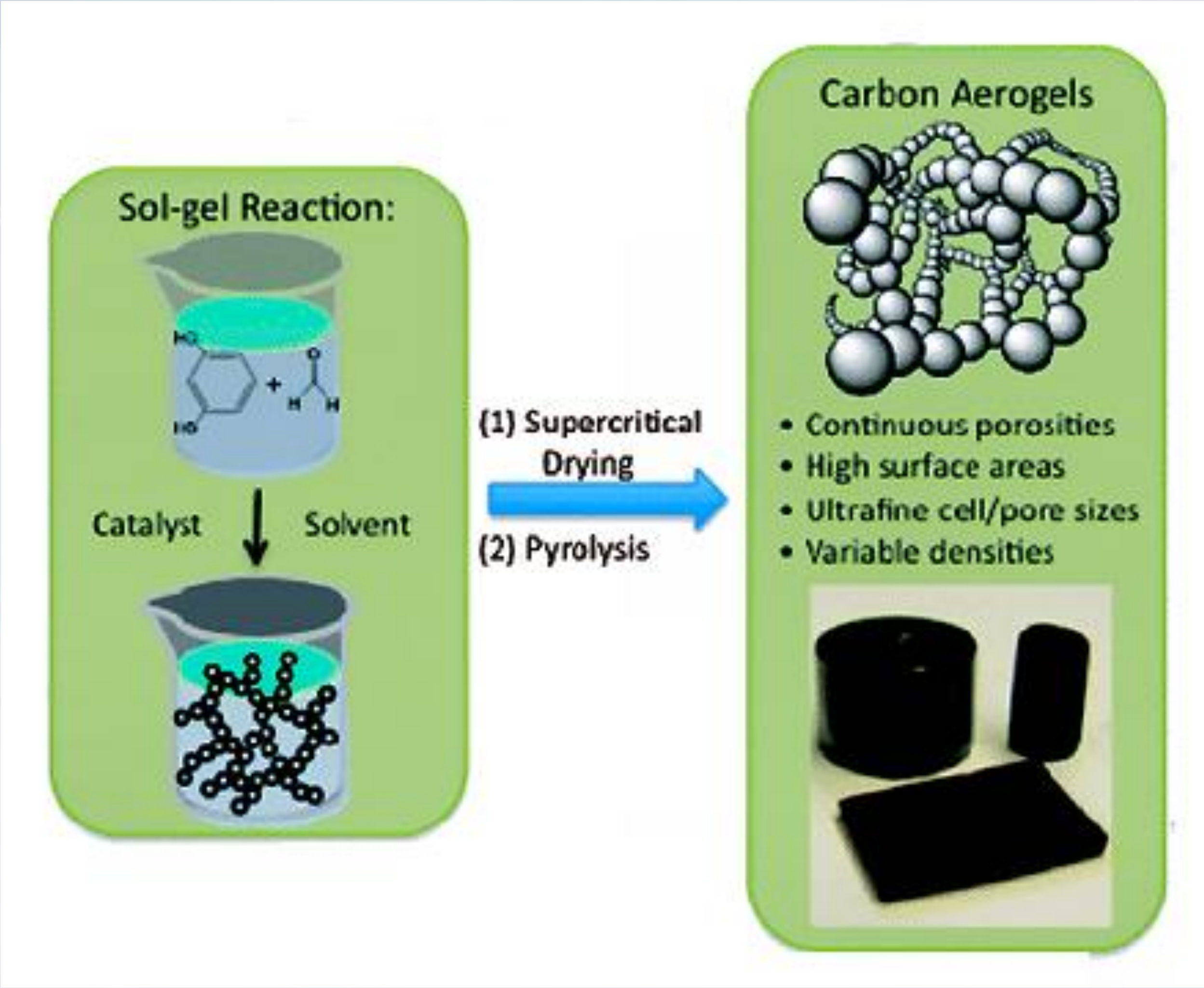
RAMAN microscopy map

based on the GO peak at 1345 cm^{-1}



Longo S., Mauro M., Daniel C., Musto P., Guerra G., *Submitted ACS Appl. Mater. Interfaces*

Carbon aerogels



Carbon aerogels are exciting materials because they not only have surface areas ranging from about 500 to 2 500 $m^2\ g^{-1}$

H.D,Gesser; P.C.,Goswami;*Chem.Rev* **1989**,89,765-788