The Use of a C-3 Building Block for Preparing New Chemicals for Different Applications

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Items of the presentation

- The ISCaMaP Research Group @ PoliMi
- A C-3 building block
- Valorization:
  - functionalization of carbon allotropes
  - new polymers
  - sulphur based crosslinking of diene elastomers

Department of Chemistry Materials and Chemical Engineering

G. Natta

Research on sustainable chemistry

Department of Chemistry Materials and Chemical Engineering

G. Natta

ISCaMaP

Innovative Sustainable Chemistry and Materials and Proteomics Group

Renewable Resources

From the European Parliament

"The recent experience of the development
of certain renewable energy sources,
particularly biofuels from food and feed crops such as
cereals, oilseeds and sugar,
has stimulated concern that
new biorefinery processes must as far as possible
be based on non-competing wastes and residues
to minimise impacts on food availability and prices."

European Parliament - Science and Technology Options Assessment
"Technology options for feeding 10 billion people"
September 2013

From wastes and residues to useful products

From the European Parliament

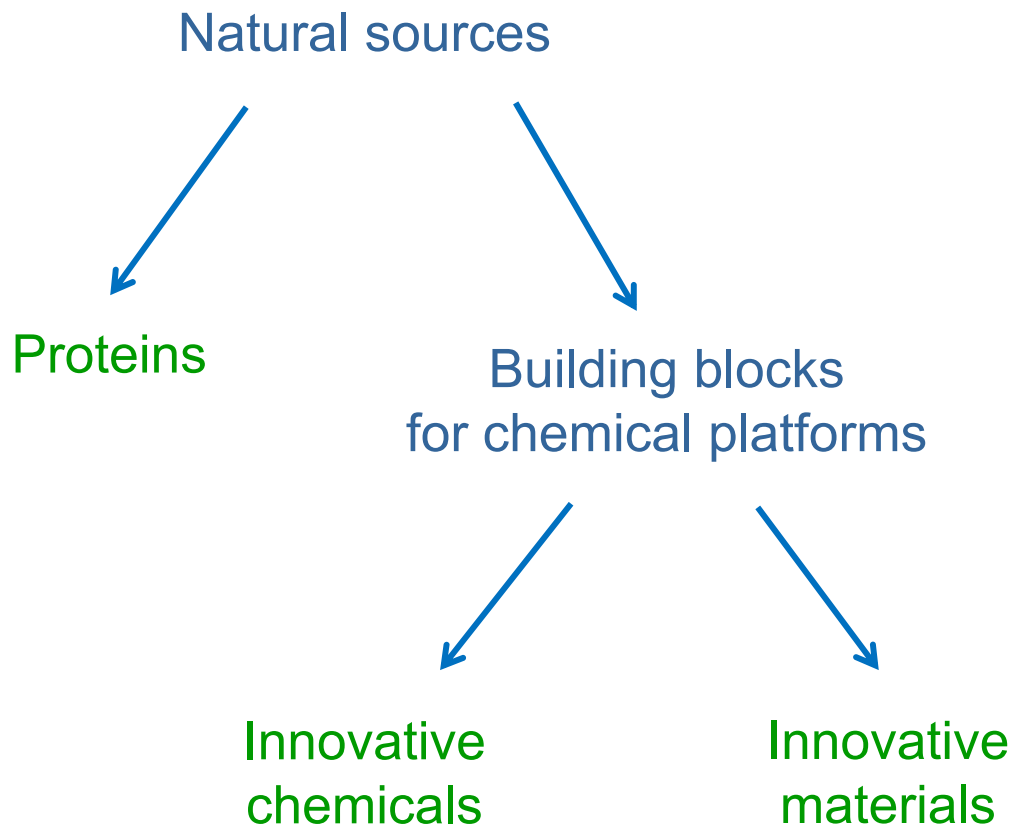
Objectives

- To transform waste and residue streams into useful products
- To identify potential markets for these products.

Waste and residue streams

"some of these materials have largely been considered a nuisance,
and a challenge for disposal without polluting the environment.
It is highly attractive therefore to be able to switch mind-set
and see such materials as useful feedstocks or raw materials."

European Parliament - Science and Technology Options Assessment
"Technology options for feeding 10 billion people"
September 2013


Natural sources
Proteins
Building blocks
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chemicals
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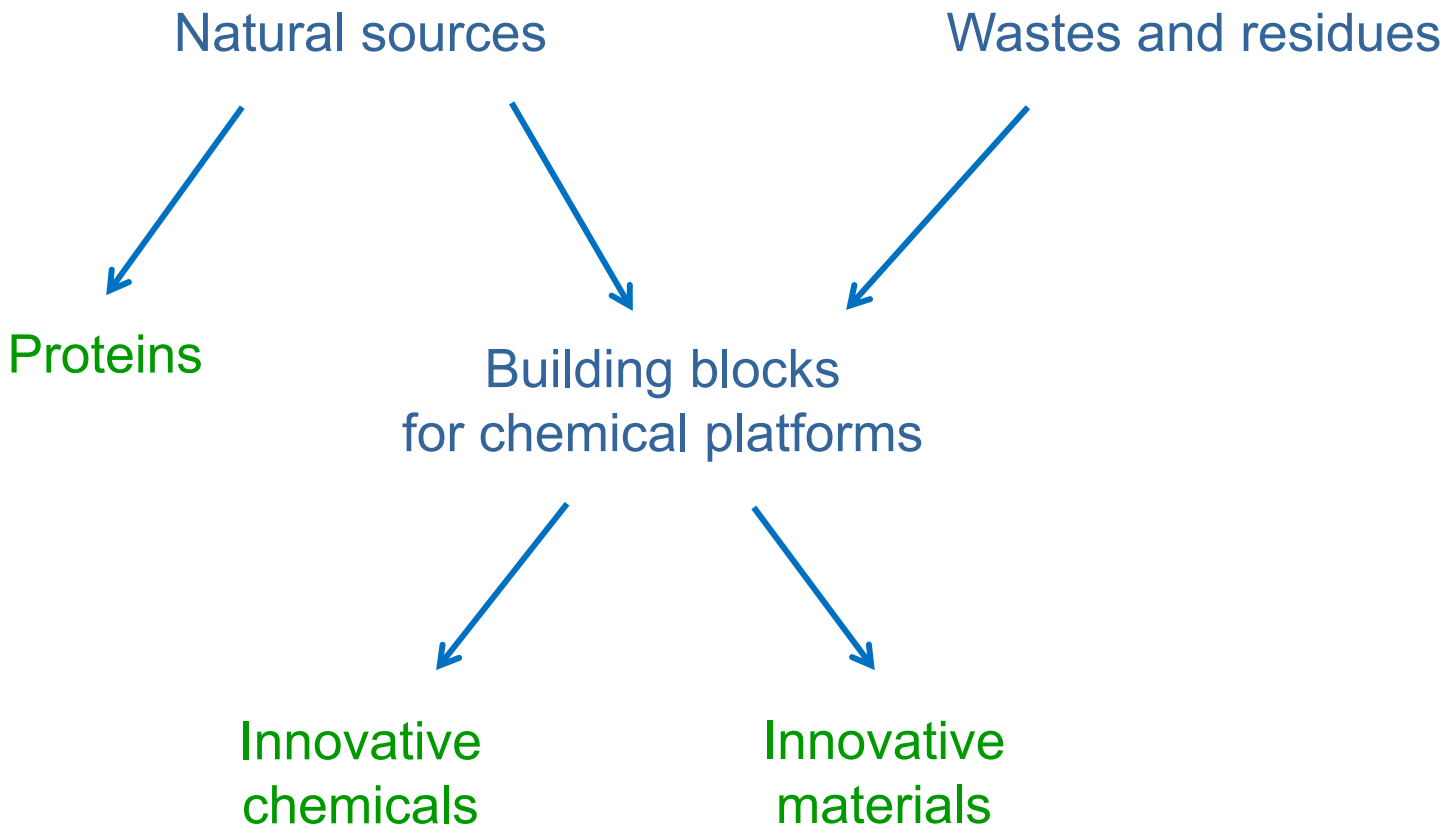
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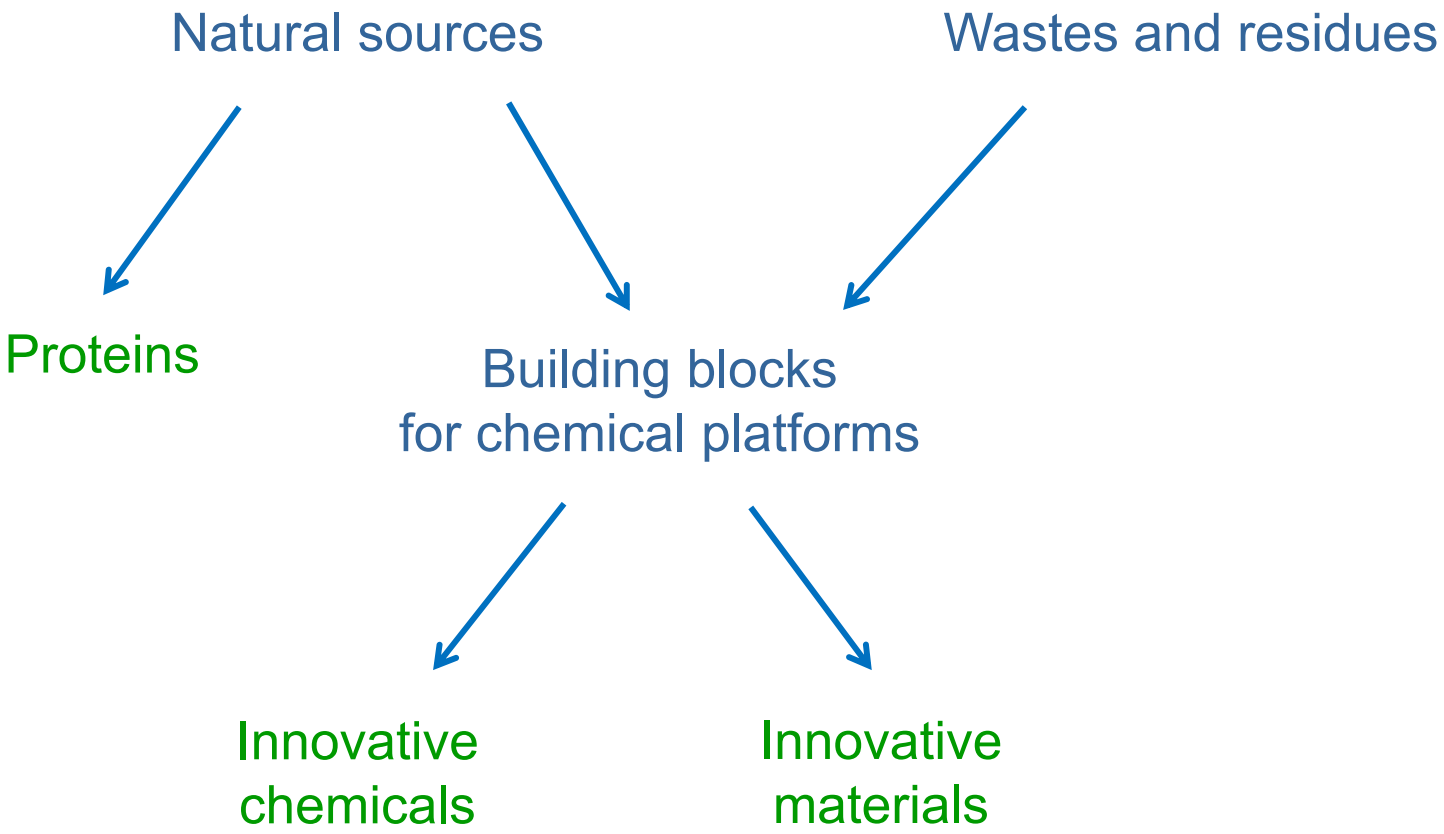


☞ Chemicals, Additives, Modifiers, Polymers

The DOE's Top Chemical Opportunities

Four Carbon 1,4-Diacids: Succinic, Fumaric and Malic

2,5-Furan dicarboxylic acid

3-Hydroxypropionic acid

Glucaric acid

Glycerol

Aspartic acid

Itaconic acid

3-Hydroxybutyrolactone

Sorbitol (Alcohol Sugar of Glucose)

Xylitol/arabinitol (Sugar alcohols from xylose and arabinose)

Glutamic acid

Levulinic acid

DOE = US Department of Energy

Criteria used in evaluating biobased product opportunities from carbohydrates

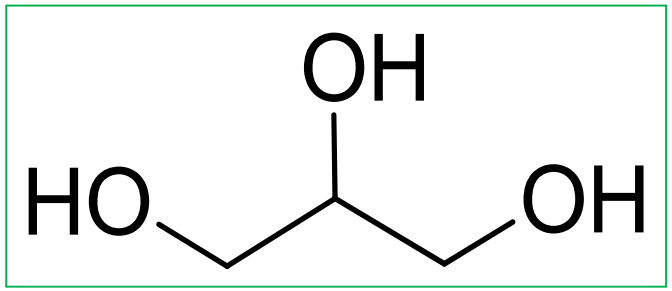
1. ***The compound or technology has received significant attention in the literature.*** A high level of reported research identifies both broad technology areas and structures of importance to the biorefinery.
2. ***The compound illustrates a broad technology applicable to multiple products.*** As in the petrochemical industry, the most valuable technologies are those that can be adapted to the production of several different structures.
3. ***The technology provides direct substitutes for existing petrochemicals.*** Products recognized by the chemical industry provide a valuable interface with existing infrastructure and utility.
4. ***The technology is applicable to high volume products.*** Conversion processes leading to high volume functional equivalents or utility within key industrial segments will have particular impact.
5. ***A compound exhibits strong potential as a platform.*** Compounds that serve as starting materials for the production of derivatives offer important flexibility and breadth to the biorefinery.
6. ***Scaleup of the product or a technology to pilot, demo, or full scale is underway.*** The impact of a biobased product and the technology for its production is greatly enhanced upon scaleup.
7. ***The biobased compound is an existing commercial product, prepared at intermediate or commodity levels.*** Research leading to production improvements or new uses for existing biobased chemicals improves their utility.
8. ***The compound may serve as a primary building block of the biorefinery.*** The petrochemical refinery is built on a small number of initial building blocks: olefins, BTX, methane, CO. Those compounds that are able to serve an analogous role in the biorefinery will be of high importance.
9. ***Commercial production of the compound from renewable carbon is well established.*** The potential utility of a given compound is improved if its manufacturing process is already recognized within the industry.

Glycerol addresses

all the criteria

Green Chem., 2010, 12, 539–554

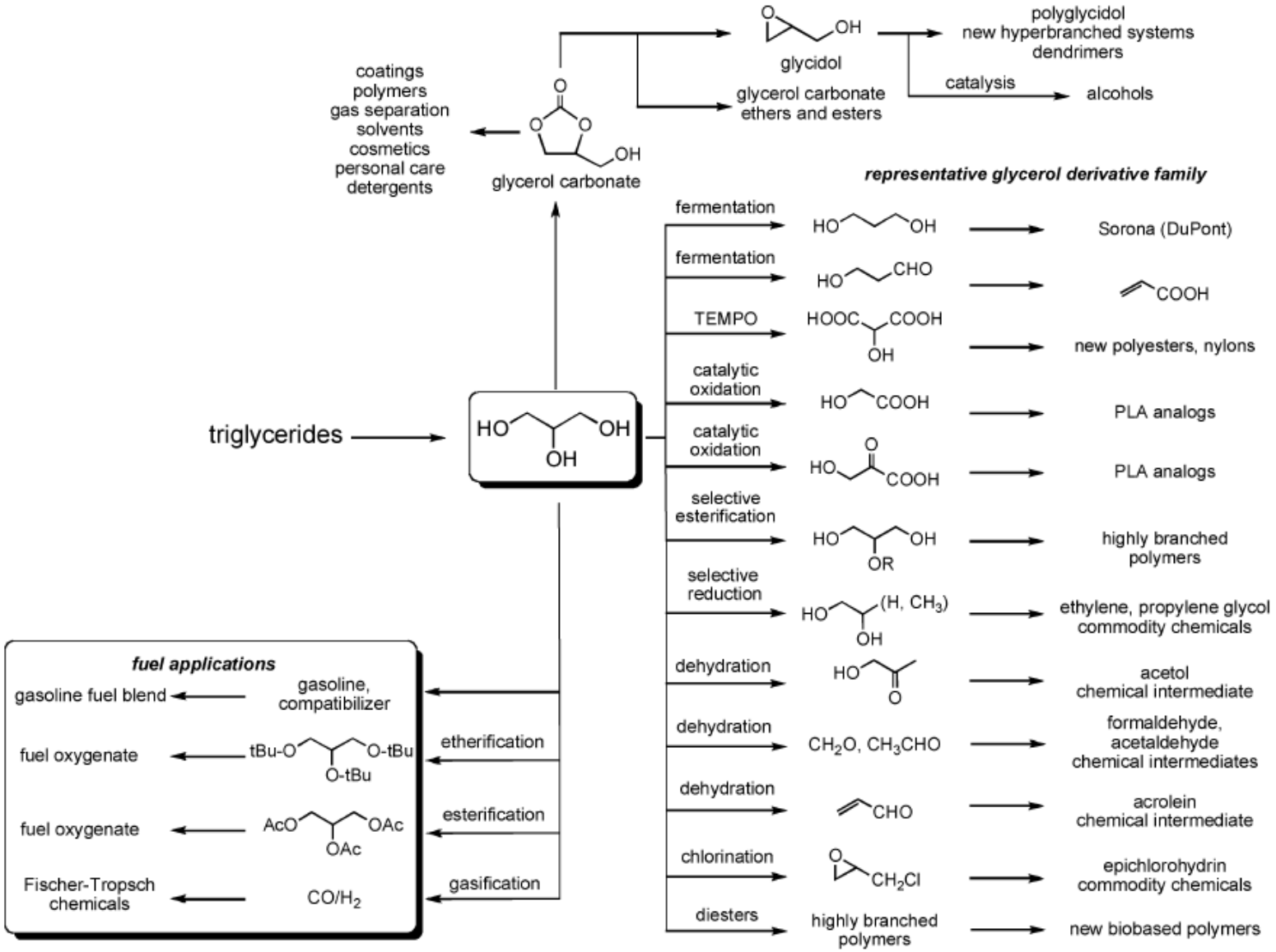
Glycerol



IUPAC: propane-1,2,3-triol
Formula: $C_3H_8O_3$
92.09 Da

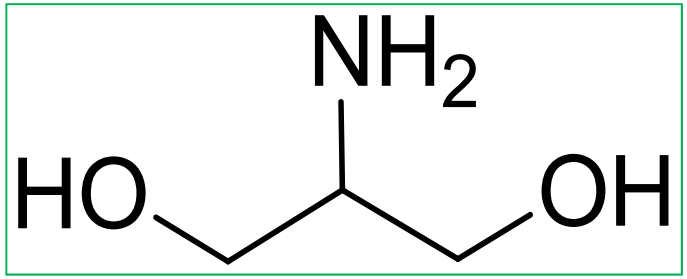
- ☞ easily available, cheap raw material
- ☞ main by-product of bio-diesel production
- ☞ not toxic
- ☞ biodegradable

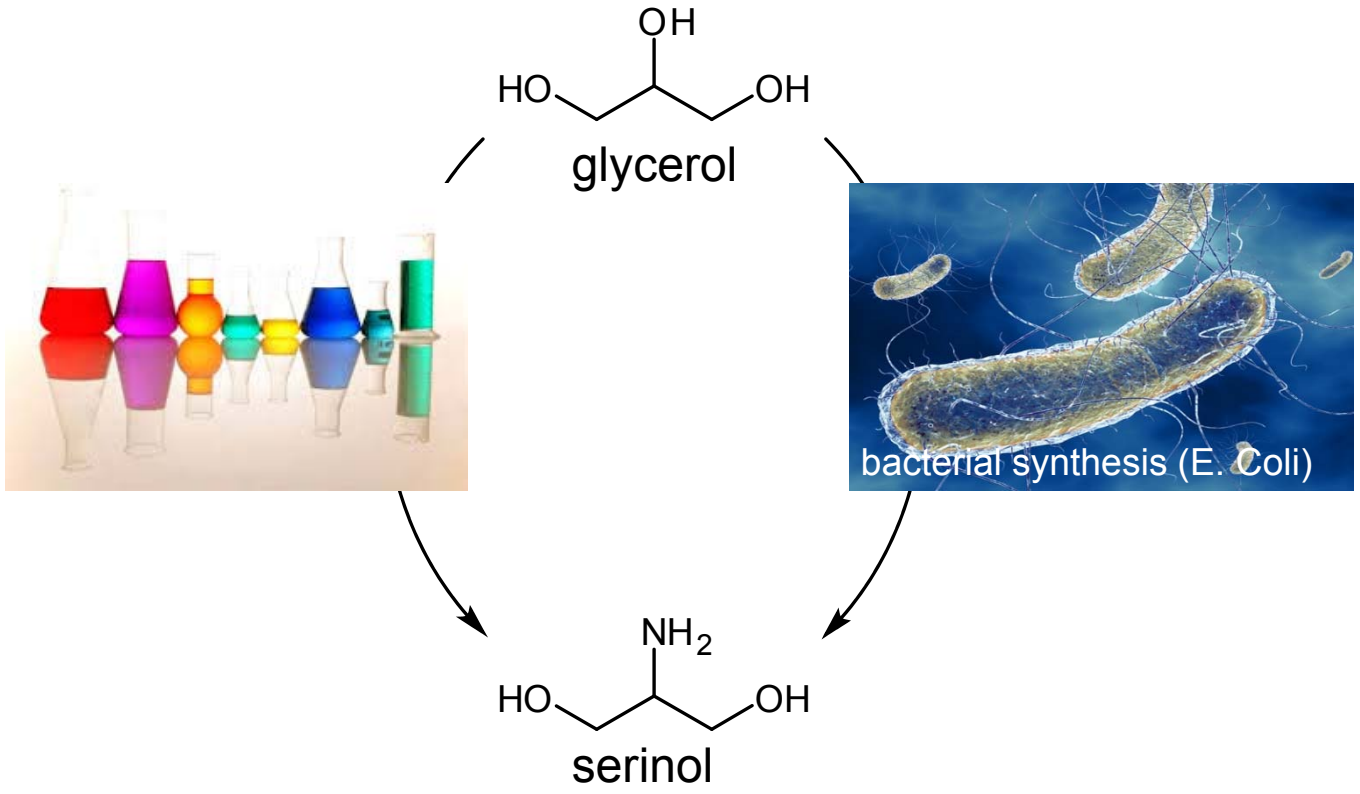
Glycerol: a primary biorefinery building block



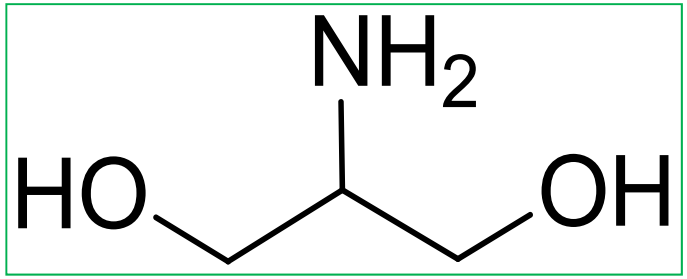
Green Chem., 2010, 12, 539–554

Selection of the building block: serinol



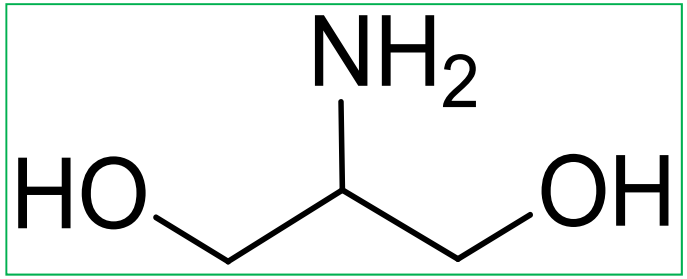


Selection of the building block: serinol



- Starting building block for many reaction pathways: many derivatives
- Chemoselectivity

Selection of the building block: serinol

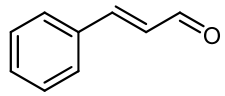
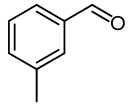
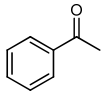
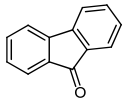


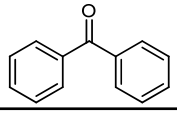
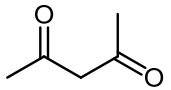
- Starting building block for many reaction pathways: many derivatives
- Chemoselectivity

↓

Reactions of the amino group with carbonyl compounds

Reaction of serinol with carbonyl compounds

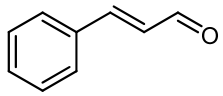
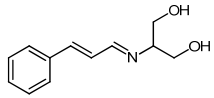
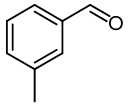
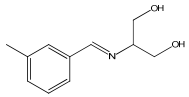
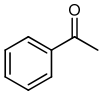
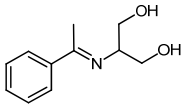
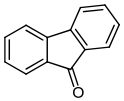
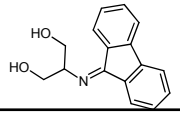
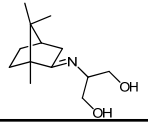
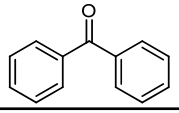
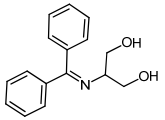
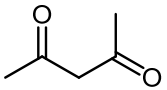
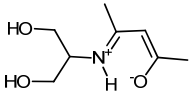
Carbonyl Compound





☞ Aromatic and sterically hindered carbonyl compounds

M. Galimberti, R. Sebastiano, V. Barbera, V. Cipolletti, G. Leonardi, S. Sun, L. Rossiello WO 2017/115253 A1

Imines from the Reaction of serinol with carbonyl compounds

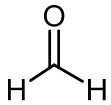
Carbonyl Compound	Product	Yield (%)
		92
		98
		83
		80
		70
		75
		95

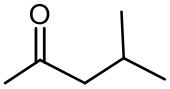
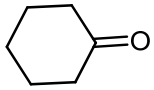
Imines

☞ Aromatic and sterically hindered carbonyl compounds lead to Imines

M. Galimberti, R. Sebastiano, V. Barbera, V. Cipolletti, G. Leonardi, S. Sun, L. Rossiello WO 2017/115253 A1

Reaction of serinol with carbonyl compounds

Carbonyl Compound


☞ Aliphatic carbonyl compounds with low steric hindrance

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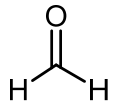
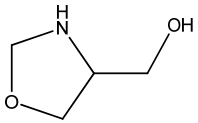
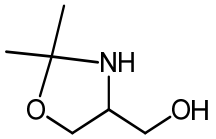
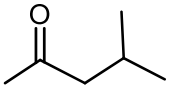
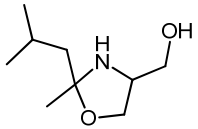
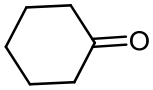
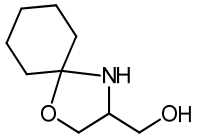
Oxazolidines from the Reaction of serinol with carbonyl compounds

Carbonyl Compound	Product	Yield (%)
		56
		90
		95
		90

Oxazolidines

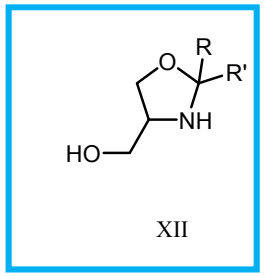
☞ Aliphatic carbonyl compounds with low steric hindrance lead to oxazolidines

M. Galimberti, R. Sebastiano, V. Barbera, V. Cipolletti, G. Leonardi, S. Sun, L. Rossiello WO 2017/115253 A1

Reaction of serinol with carbonyl compounds.

Without steric hindrance

and aromatic substituents

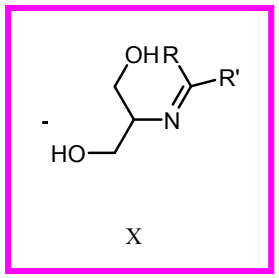


XII

Oxazolidines

With steric hindrance

and aromatic substituents



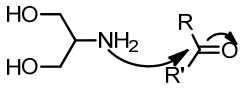
X

Imines

M. Galimberti, R. Sebastiano, V. Barbera, V. Cipolletti, G. Leonardi, S. Sun, L. Rossiello WO 2017/115253 A1

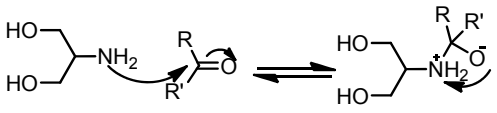
Reaction of serinol with carbonyl compounds – Hypothesis of a mechanism

The nucleophilic nitrogen reacts with carbonyl group



Reaction of serinol with carbonyl compounds – Hypothesis of a mechanism

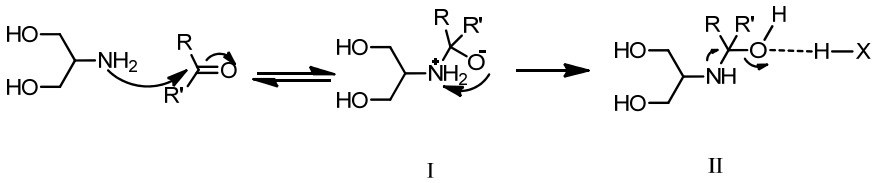
Proton transfer



I

Reaction of serinol with carbonyl compounds – Hypothesis of a mechanism

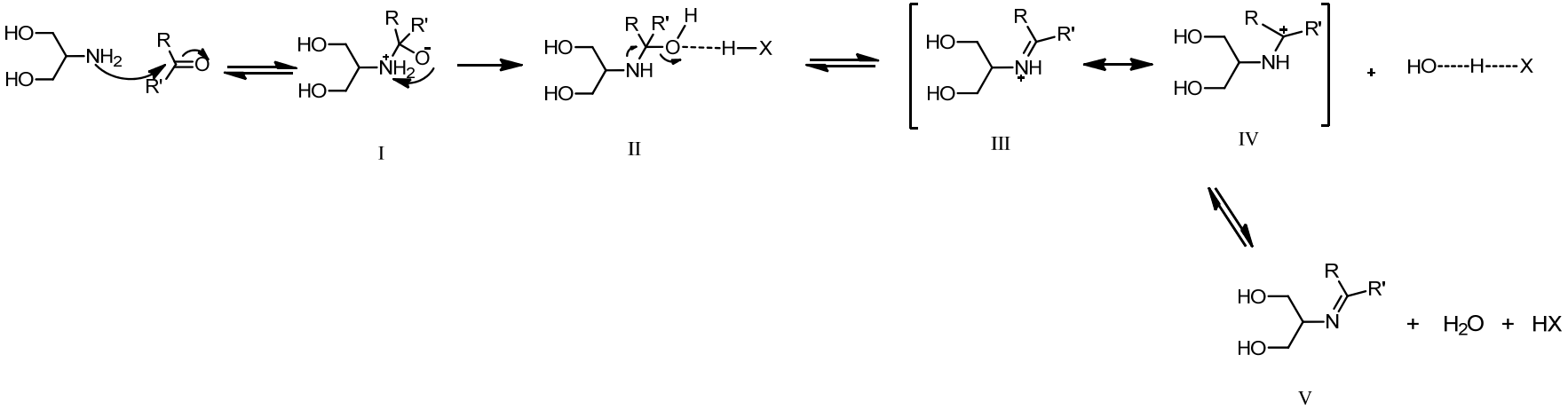
Release of a water molecule



I II

H—X = General or specific acid cat.

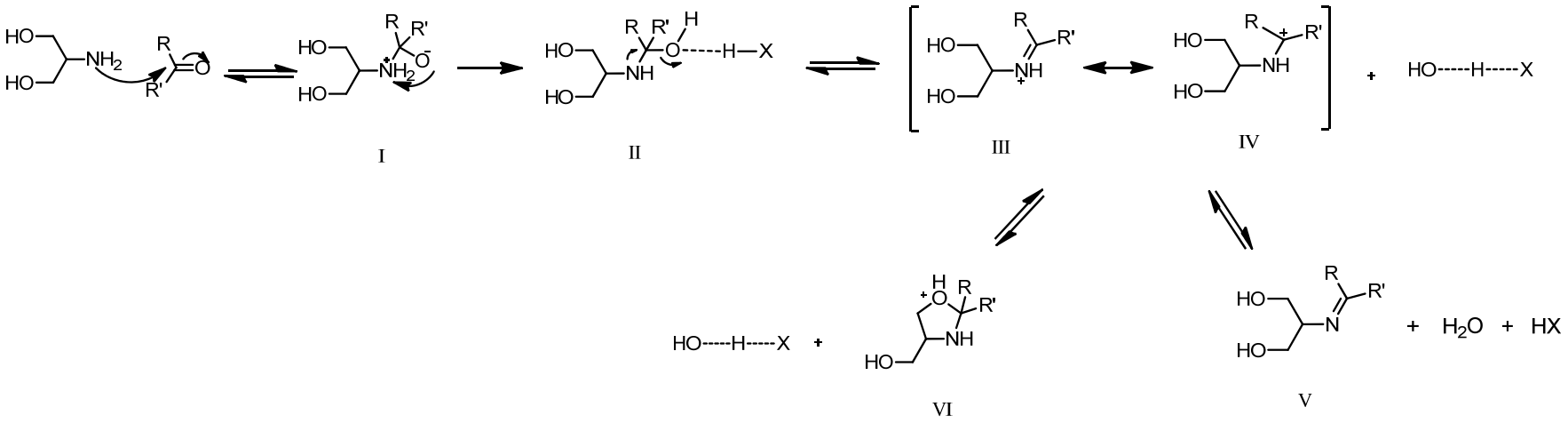
Reaction of serinol with carbonyl compounds – Hypothesis of a mechanism



Proton abstraction. Formation of an imine

H—X = General or specific acid cat.

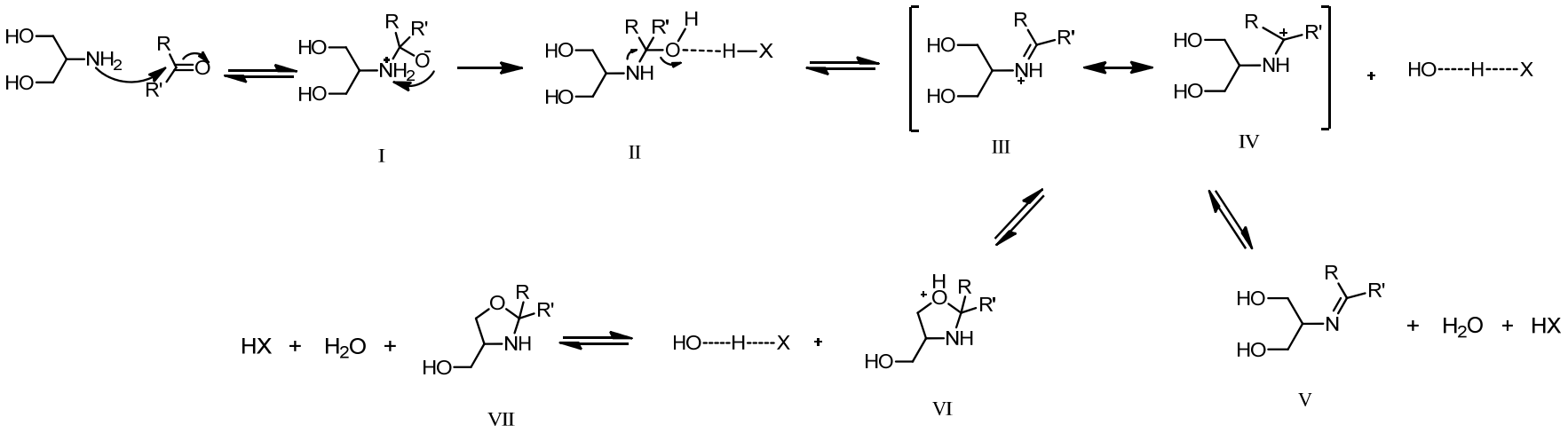
Reaction of serinol with carbonyl compounds – Hypothesis of a mechanism



Nucleophilic intramolecular addition: protonated oxazolidine

H—X = General or specific acid cat.

Reaction of serinol with carbonyl compounds – Hypothesis of a mechanism



Proton abstraction

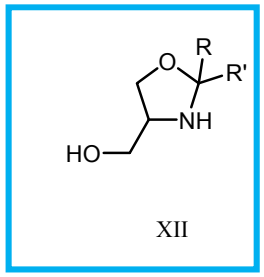
H—X = General or specific acid cat.

Functionalization of sp^2 carbon allotropes

Reaction of serinol with carbonyl compounds.

Without steric hindrance

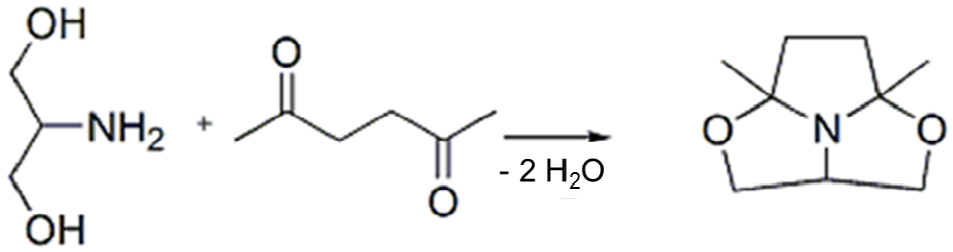
and aromatic substituents



XII

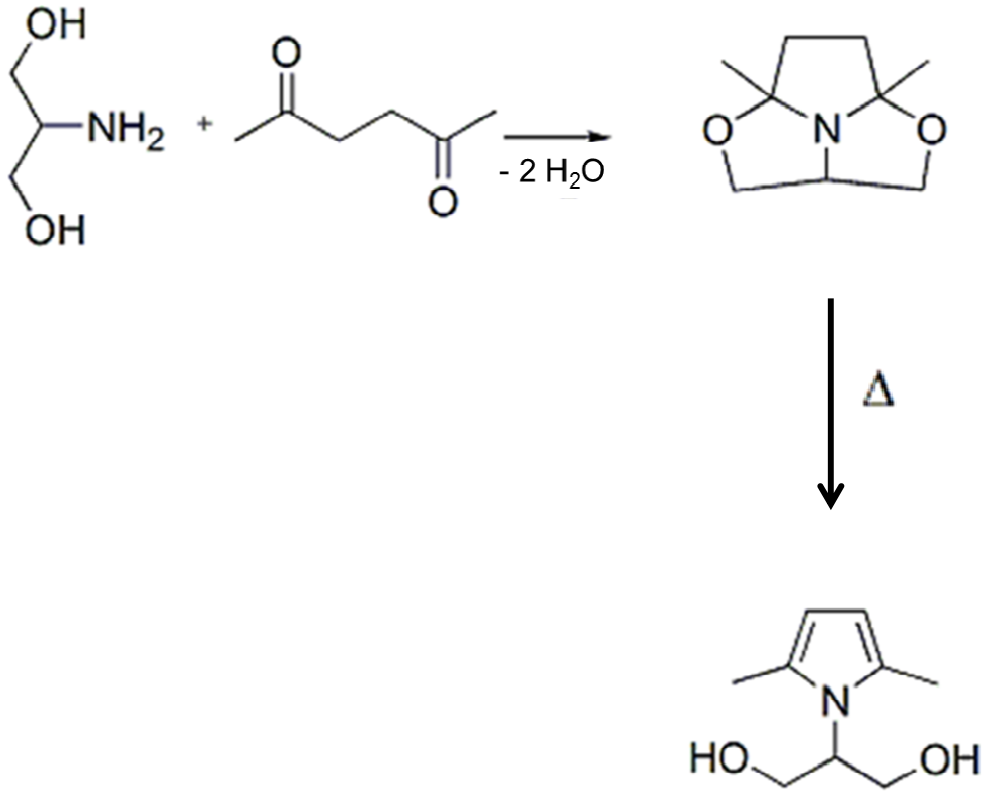
Oxazolidines

Reaction of serinol with dicarbonyl compound



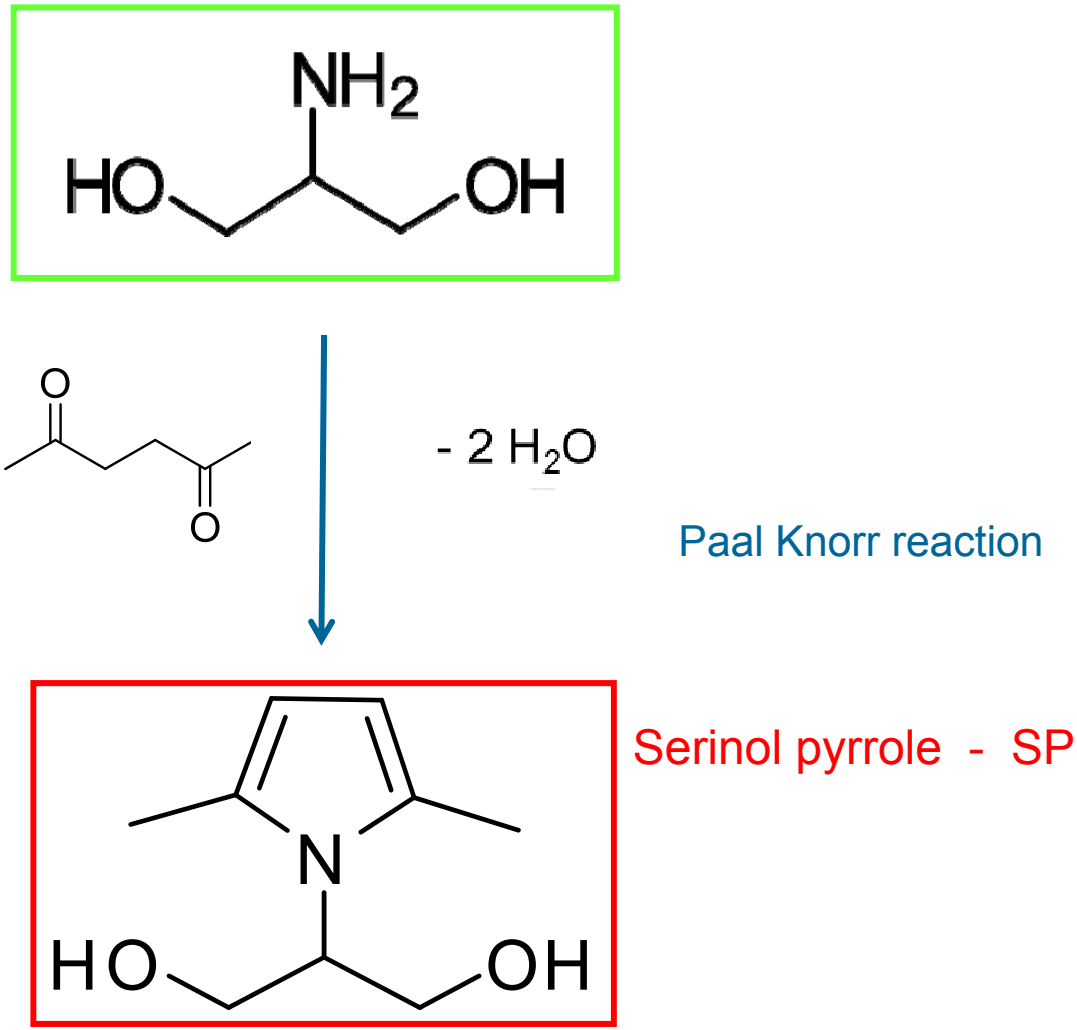
M. Galimberti, V. Barbera, A. Citterio, R. Sebastiano, A. Truscello, A. M. Valerio, L. Conzatti, R. Mendichi, *Polymer*, vol 63, 20 April 2015, Pages 62–70
M. Galimberti, V. Barbera, S. Guerra, L. Conzatti, C. Castiglioni, L. Brambilla, A. Serafini,, *RSC Adv.*, 2015, *5*, 81142-81152 DOI: 10.1039/C5RA11387C
V. Barbera, S. Musto, A. Citterio, L. Conzatti, M. Galimberti,, *eXPRESS Polymer Letters* 2016, *10* (7) 548–558

Reaction of serinol with dicarbonyl compound



M. Galimberti, V. Barbera, A. Citterio, R. Sebastiano, A. Truscello, A. M. Valerio, L. Conzatti, R. Mendichi, *Polymer*, vol 63, 20 April 2015, Pages 62–70
M. Galimberti, V. Barbera, S. Guerra, L. Conzatti, C. Castiglioni, L. Brambilla, A. Serafini,, *RSC Adv.*, 2015, *5*, 81142-81152 DOI: 10.1039/C5RA11387C
V. Barbera, S. Musto, A. Citterio, L. Conzatti, M. Galimberti,, *eXPRESS Polymer Letters* 2016, *10* (7) 548–558

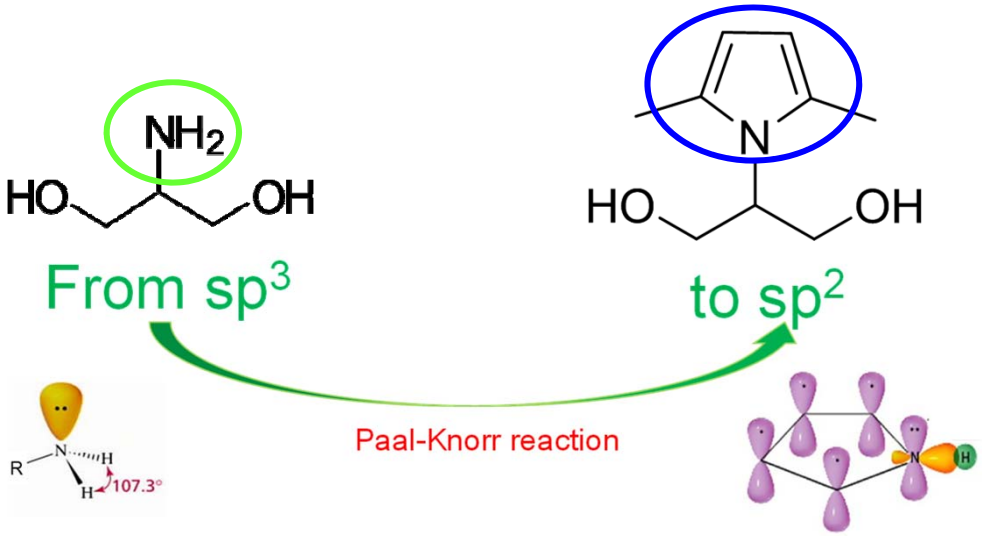
Oxidation of CA with pyrrole derivative



2-(2,5-dimethyl-1*H*-pirrol-1-yl) -1,3-propanediol

Barbera, V., Citterio, A., Galimberti, M., Leonardi, G., Sebastiano, R., Shisodia, S.U., Valerio A.M. WO 2015 189411 A1

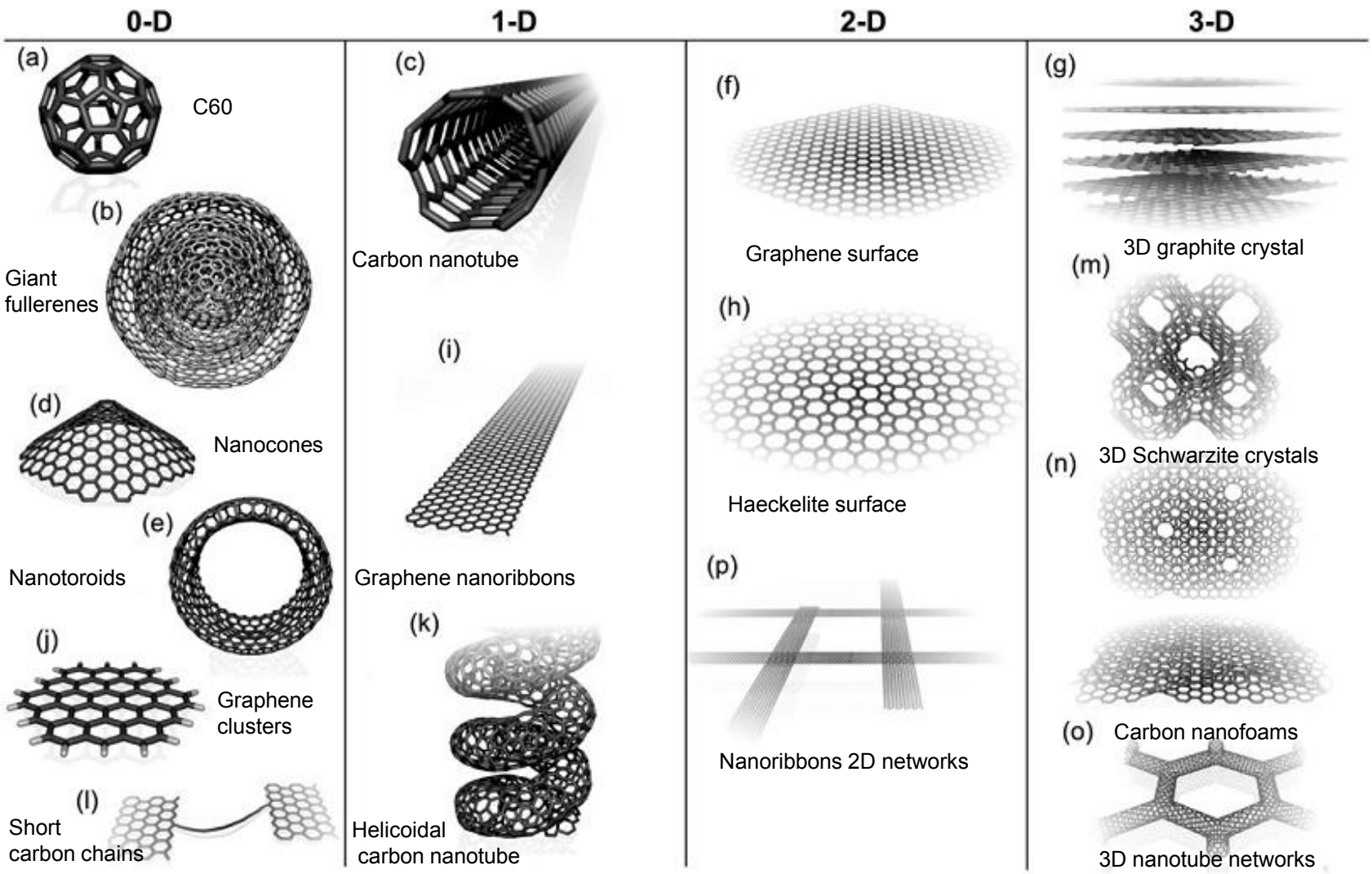
Neat synthesis of Serinol pyrrole



- Yield: at least 96%
- Atom efficiency: 85%
- Easy procedure
- No solvent
- By product: H_2O

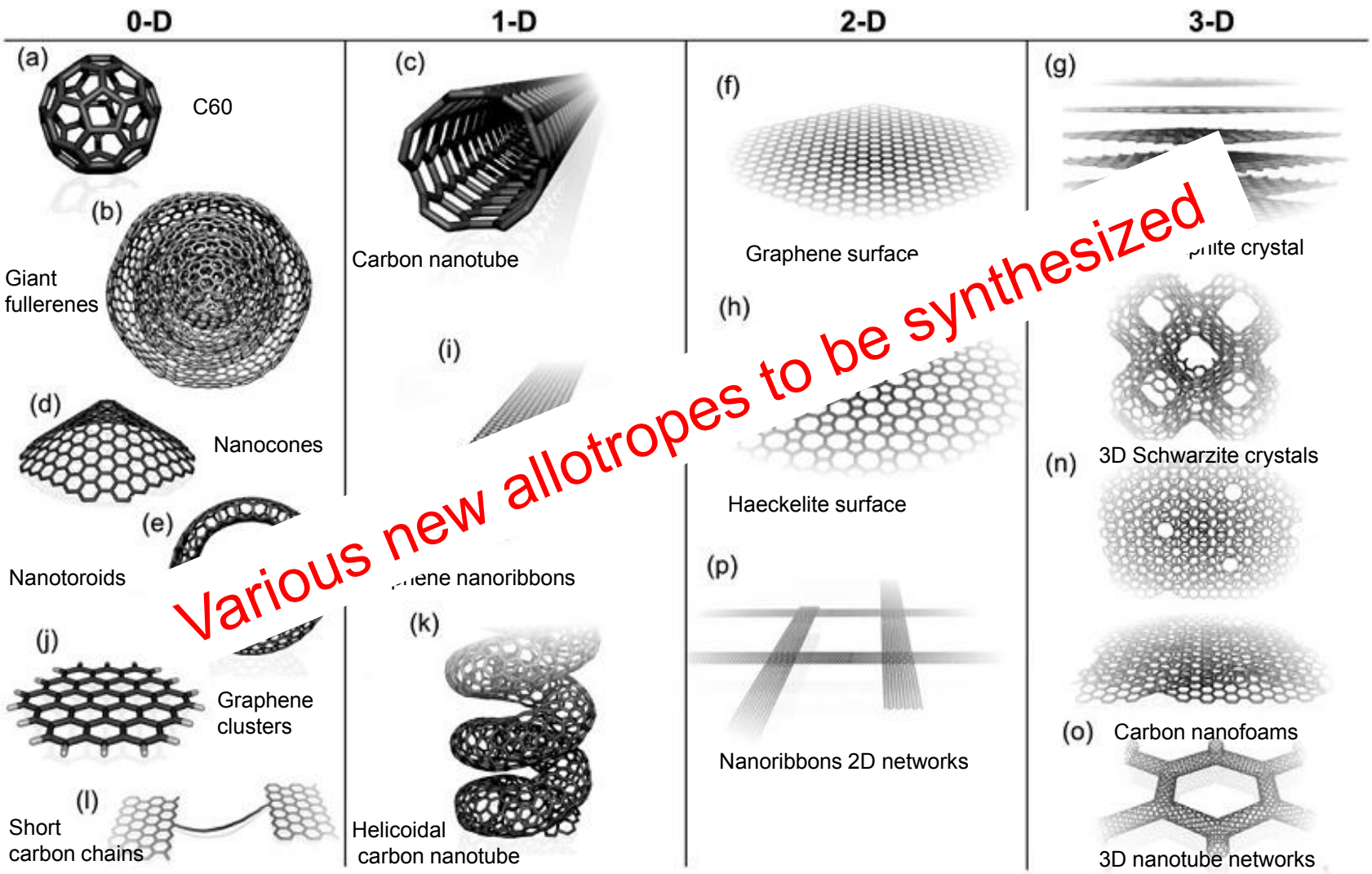
Barbera, V., Citterio, A., Galimberti, M., Leonardi, G., Sebastiano, R., Shisodia, S.U., Valerio A.M. WO 2015 189411 A1
Galimberti, M., Barbera, V., Guerra, S., Conzatti, L., Castiglioni, C., Brambilla, L., A. Serafini, RSC Advances, 5(99), (2015) 81142-81152

sp^2 carbon allotropes



M. Terrones, et al. Nano Today 5 (4) (2010) 351e372.

sp^2 carbon allotropes

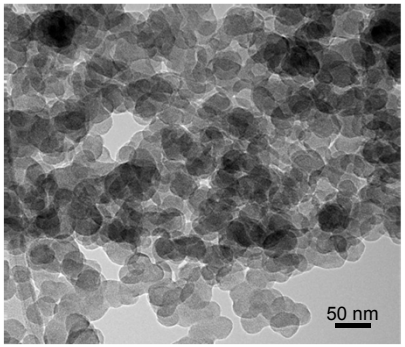


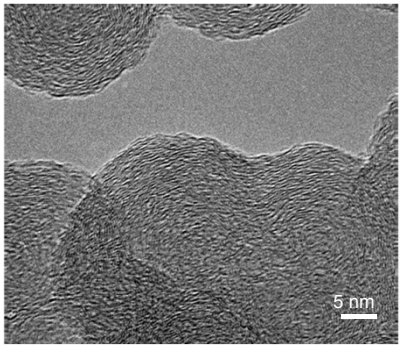
M. Terrones, et al. Nano Today 5 (4) (2010) 351e372.

Jin Zhang et al, Carbon 98 (2016) 708e732

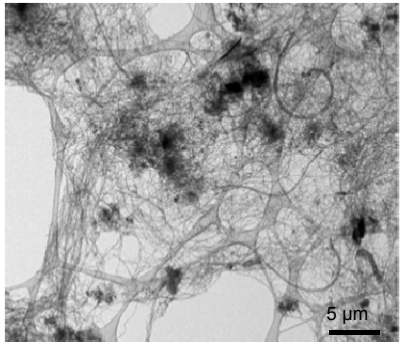
Carbon allotropes

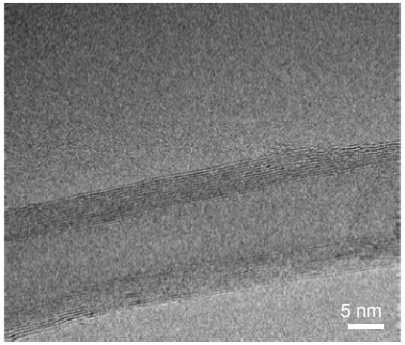
CB



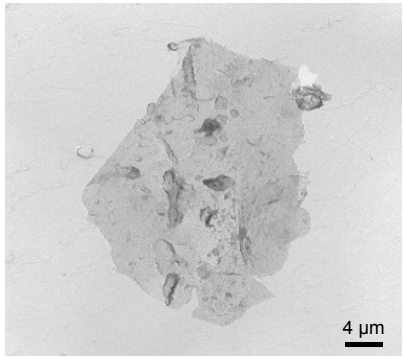


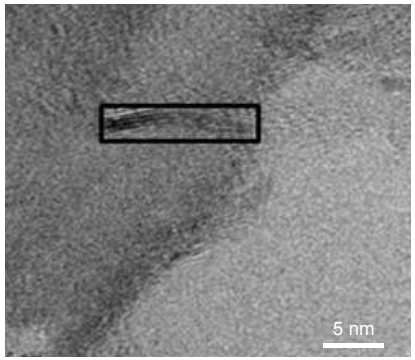
CNT





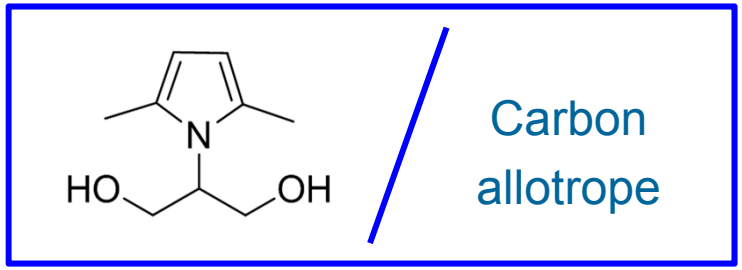
FLG



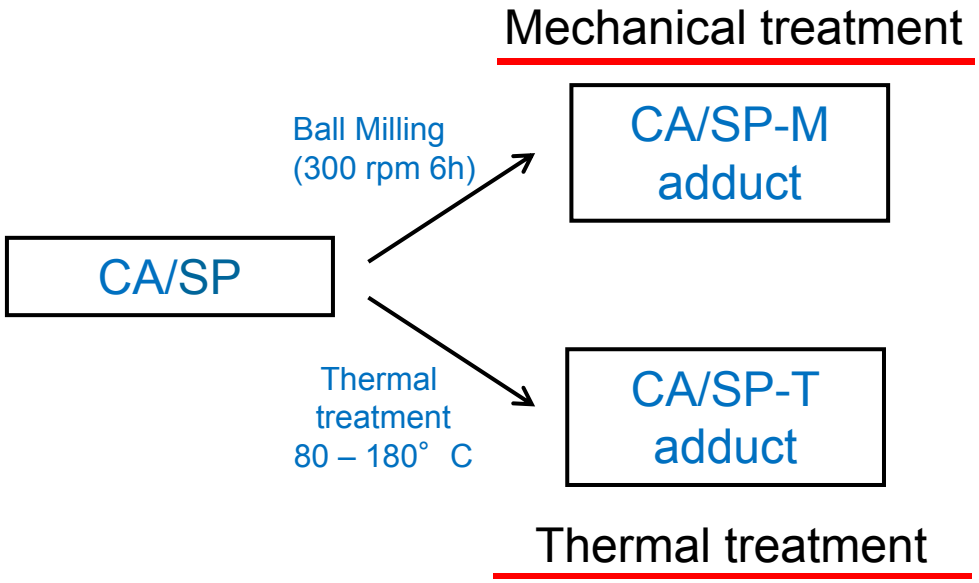


High-resolution
transmission electron microscopy
(HR-TEM)

Adducts of SP with CA - Preparation

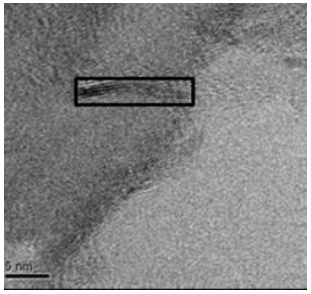
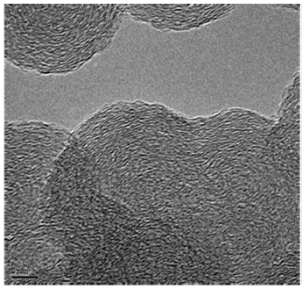
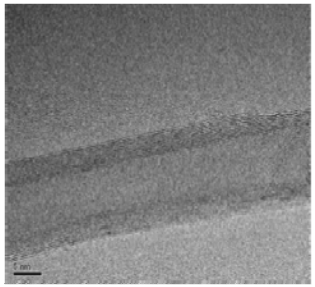


CA-SP



Barbera, V., Citterio, A., Galimberti, M., Leonardi, G., Sebastiano, R., Shisodia, S.U., Valerio A.M. WO 2015 189411 A1
Galimberti, M., Barbera, V., Guerra, S., Conzatti, L., Castiglioni, C., Brambilla, L., A. Serafini, RSC Advances, 5(99), (2015) 81142-81152

High yield functionalization!

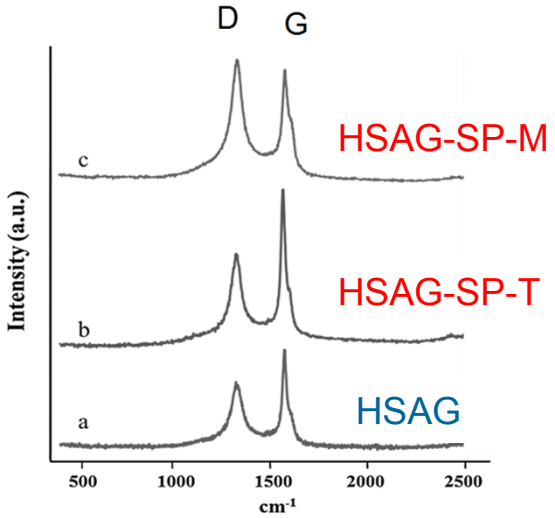
BET Surface area: [m^2/g]	**300**	**77**	**275**
Initial functional groups: [mmol/g]	**1.7**	**0.9**	**2.0**
Yields (%)*:	**96**	**82**	**92**

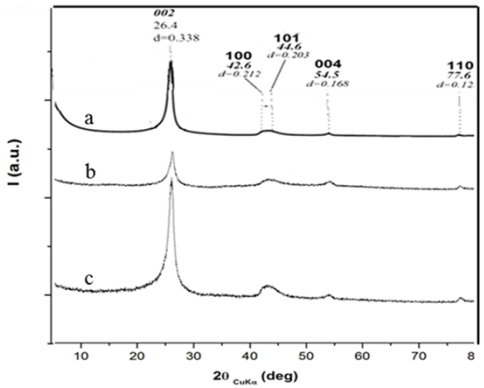
*was estimated through:

$$\text{Functionalization Yield (\%)} = 100 * \frac{\text{SP mass \% in (CA–SP adduct) after acetone washing}}{\text{SP mass \% in (CA–SP adduct) before acetone washing}}$$

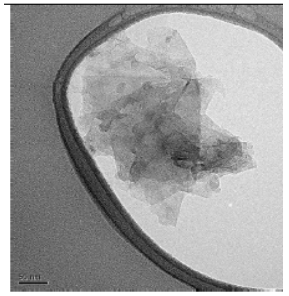
Galimberti, M., Barbera, V., Sebastiano, R., Citterio, A., Leonardi, G. WO/2016/050887 A1 (2016)

Adducts of SP with HSAG





Few layers graphene



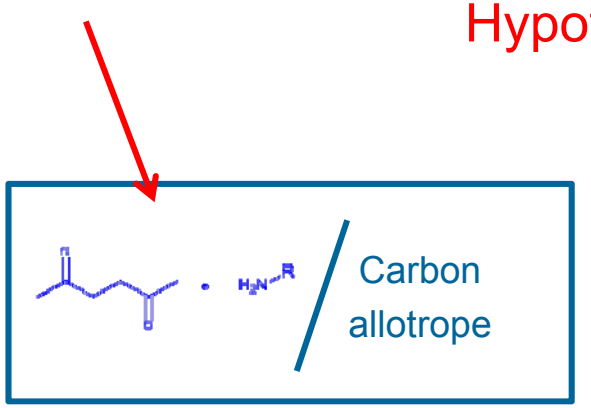
From water suspension

Results from elemental, TGA, IR, XPS, Raman, XRD, HRTEM analysis

Galimberti, M., Barbera, V., Sebastiano, R., Citterio, A., Leonardi, G. WO/2016/050887 A1 (2016)
Galimberti, M., Barbera, V., Guerra, S., Conzatti, L., Castiglioni, C., Brambilla, L., A. Serafini, RSC Advances, 5(99), (2015) 81142-81152

Facile functionalization of graphene layers (carbon materials)

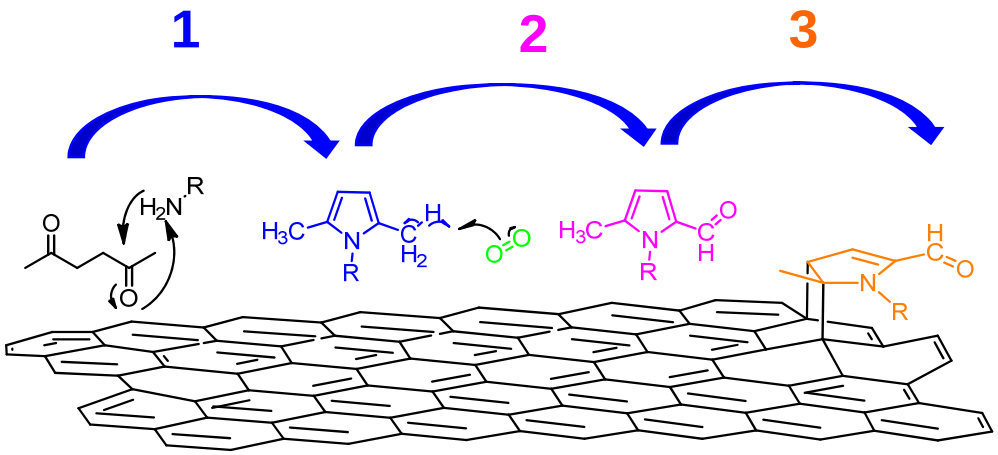
Hypothesis for the mechanism



Paal – Knorr Reaction

Carbocatalyzed Oxidation

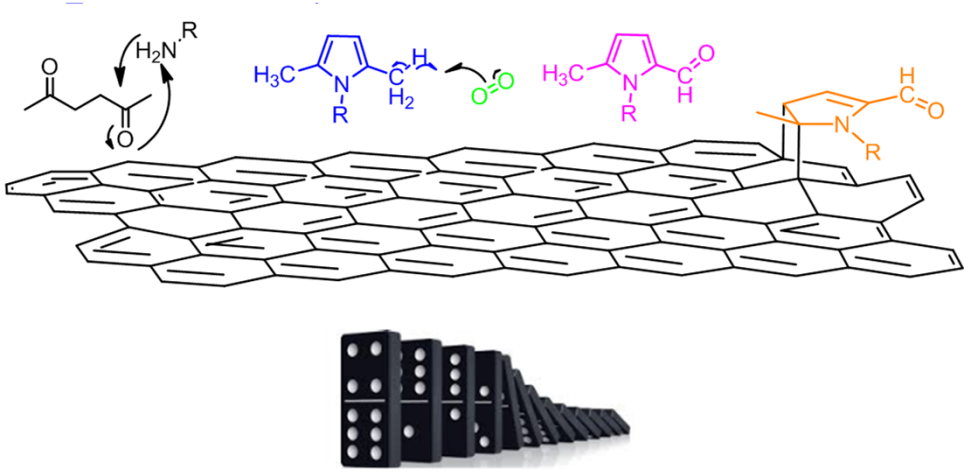
Diels-Alder reaction



Barbera, V., Milani, A., Brambilla, I., Castiglioni, C., Palazzolo, A., Galimberti M., Presentation at AICING 2017, Milano June 2017; submitted for publication

Thanks to the carbon allotrope!

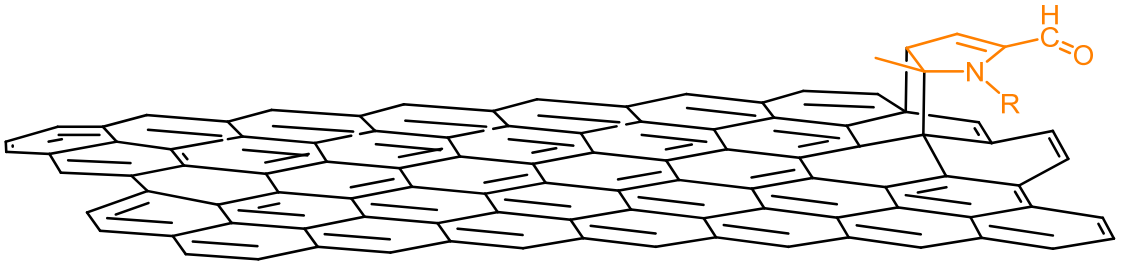
- Support: absorption of pyrrole ring thanks to π-π interaction
- Oxidation catalyst: protection of pyrrole ring and oxidation of lateral substituent
- Substrate for the cycloaddition reaction, i.e. for functionalization



Barbera, V., Milani, A., Brambilla, I., Castiglioni, C., Palazzolo, A., Galimberti M., Presentation at AICING 2017, Milano June 2017; submitted for publication

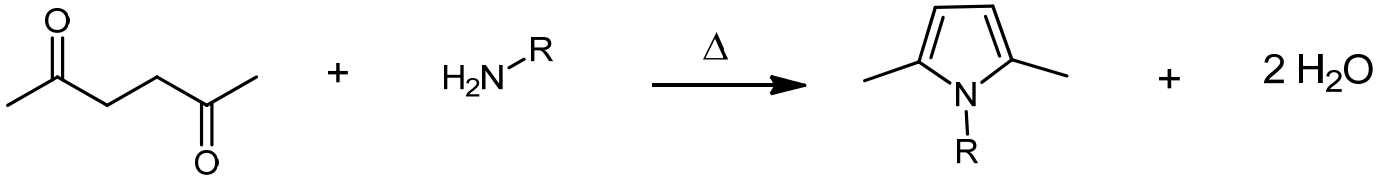
Adducts of SP with HSAG

- Functional groups up to 20%
- In plane order substantially unaltered
- No expansion of interlayer distance



Galimberti, M., Barbera, V., Guerra, S., Conzatti, L., Castiglioni, C., Brambilla, L., A. Serafini, RSC Advances, 5(99), (2015) 81142-81152

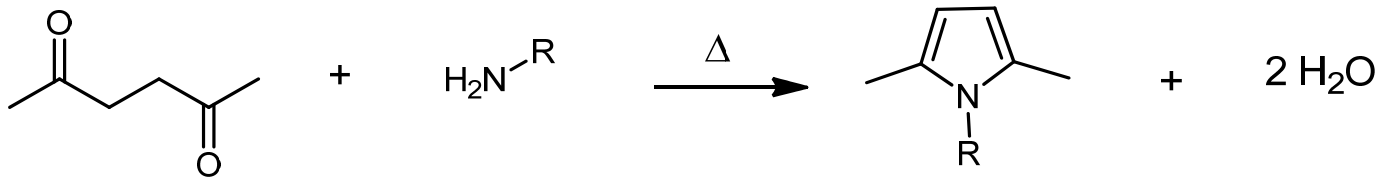
Pyrrole compounds from neat Paal Knorr reaction

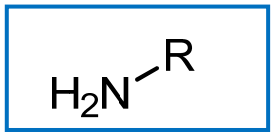


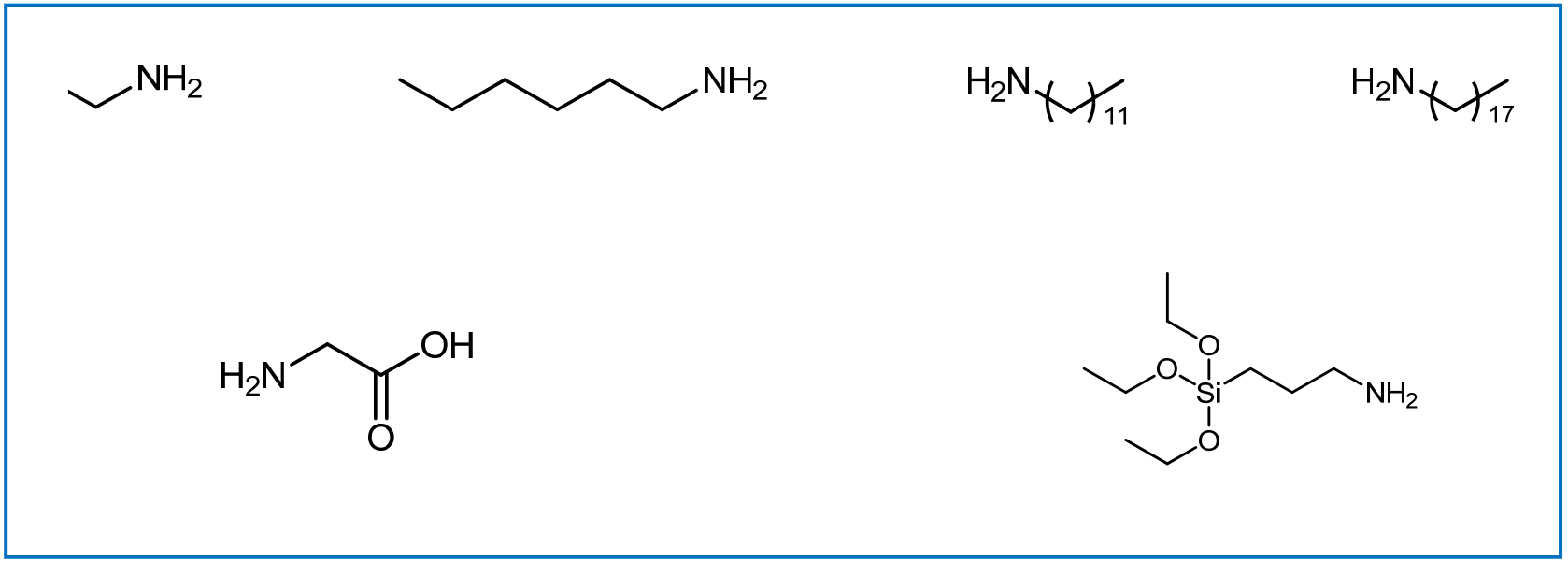
M. Galimberti, V. Barbera, S. Guerra, A. Bernardi, Facile functionalization of sp^2 carbon allotropes with a biobased Janus molecule. *Rubber Chemistry and Technology,* 2017, 90(2), 285-307.

V. Barbera, A. Bernardi, A. Palazzolo, A. Rosengart, L. Brambilla, M. Galimberti, Facile and sustainable functionalization of graphene layers with pyrrole compounds *Pure and Applied Chemistry*, 2017 | DOI: https://doi.org/10.1515/pac-2017-0708

Pyrrole compounds from neat Paal Knorr reaction

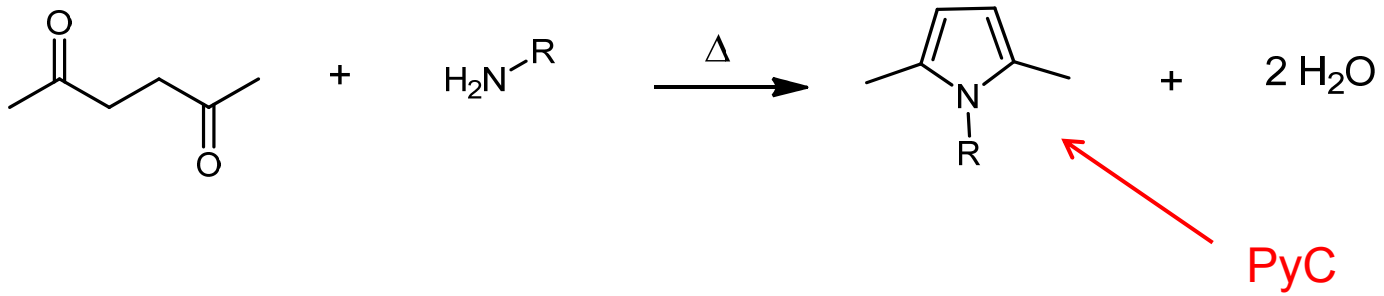




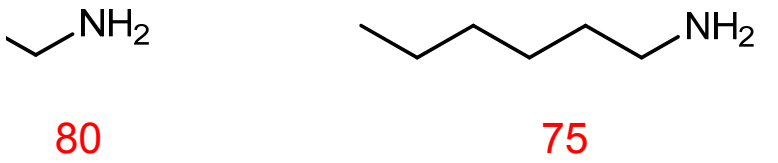


V. Barbera, A. Bernardi, A.Palazzolo, A. Rosengart, L. Brambilla, M. Galimberti Pure and Applied Chemistry

Pyrrole compounds from neat Paal Knorr reaction

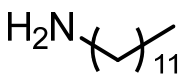


Yield %



80

75



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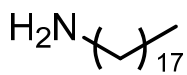

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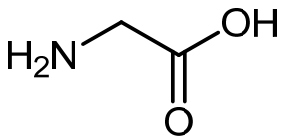

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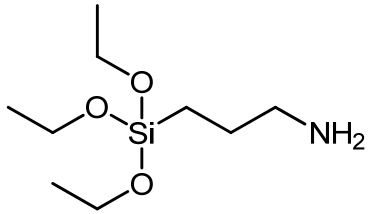
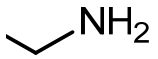


70

V. Barbera, A. Bernardi, A.Palazzolo, A. Rosengart, L. Brambilla, M. Galimberti Pure and Applied Chemistry

HSAG / PyC adducts

Functionalization Yield %

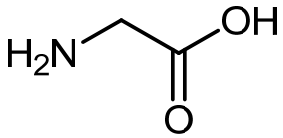


57

53

73

55



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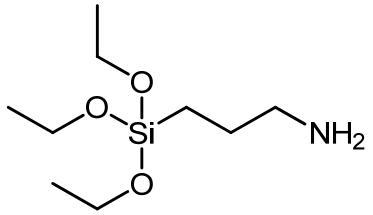
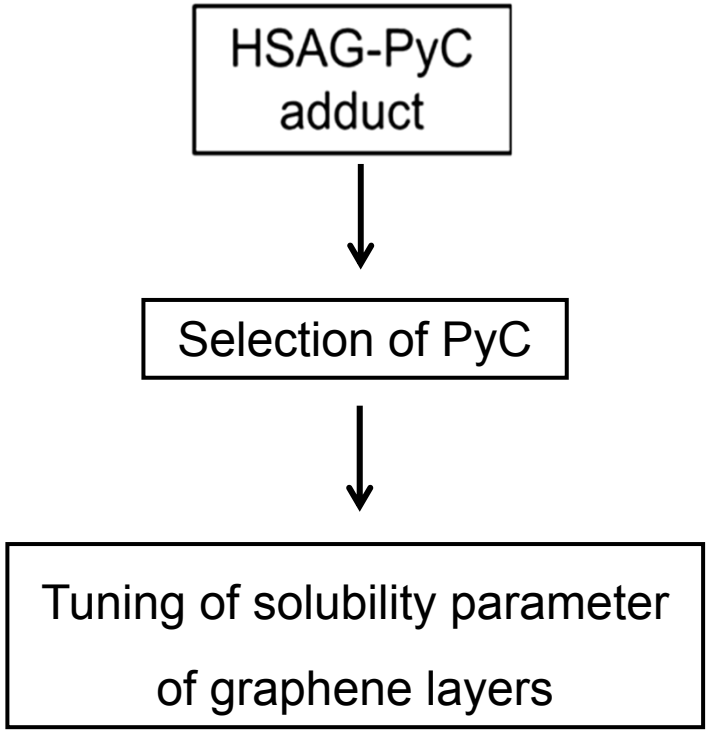


70

V. Barbera, A. Bernardi, A.Palazzolo, A. Rosengart, L. Brambilla, M. Galimberti Pure and Applied Chemistry

Tuning of solubility parameter of graphene layers



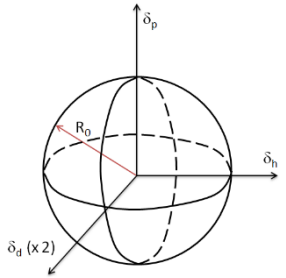
HSAG / PyC adducts - Tuning of solubility parameters

Experimental determination

Stable suspensions
in solvents
with different δ

Theoretical predictions

Computational model:
Hansen solubility parameters

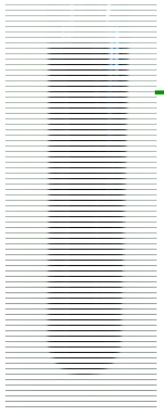


Determination of solubility parameters of HSAG-PyC - Experiments

Adduct HSAG-	solvents water	isopropanol	ethyl acetate	toluene	heptane
TMP	bad (↓)	good	good	good	good
HP	bad (↑)	bad (↓)	good	bad (↓)	good
DDcP	bad (↑)	good	good	bad (↓)	bad (↓)
SP	good	good	good	bad (↓)	bad (↓)
Gly	bad (↓)	good	good	good	bad (↓)
APTESP	bad (↑)	bad (↓)	bad (↓)	good	good

No suspension: **bad**

Unstable suspension: **bad**



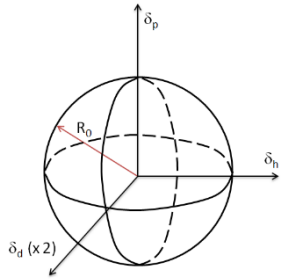
Stable suspension: **good**

V. Barbera, A. Bernardi, A.Palazzolo, A. Rosengart, L. Brambilla, M. Galimberti Paper accepted for publication on Pure and Applied Chemistry

Calculation of solubility parameters of HSAG / PyC adducts

By applying the Hansen Solubility Sphere representation of miscibility

Cohesive energy (Hildebrand model) of a substance:
sum of three contributions:
dispersion, polar, hydrogen bonding:
U_D, U_P, U_H



↓

The substance is identified by three coordinates (δ_D, δ_P and δ_H)
in the Hansen Parameters space

↓

The distance between two points (e.g. of a solute and a solvent)
is related to the cohesive energy difference

↓

Two points close to each other in the Hansen space
indicate miscible substances

Estimation of HSP of a solute *i*

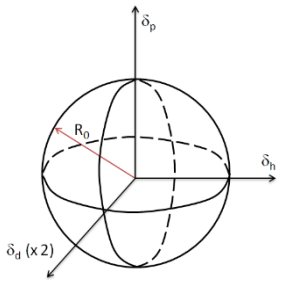
Dispersion tests are performed with different solvents *j*, distinguishing:
- good solvents, which provide stable solutions/dispersions
- bad solvents, which do not give stable dispersions.

↓

Minimization of the ratio * $\dfrac{\text{Distance between the solute and the solvent}}{R_o \text{ radius of interaction}}$

↓

Calculation of the center coordinates of the Hansen solubility sphere

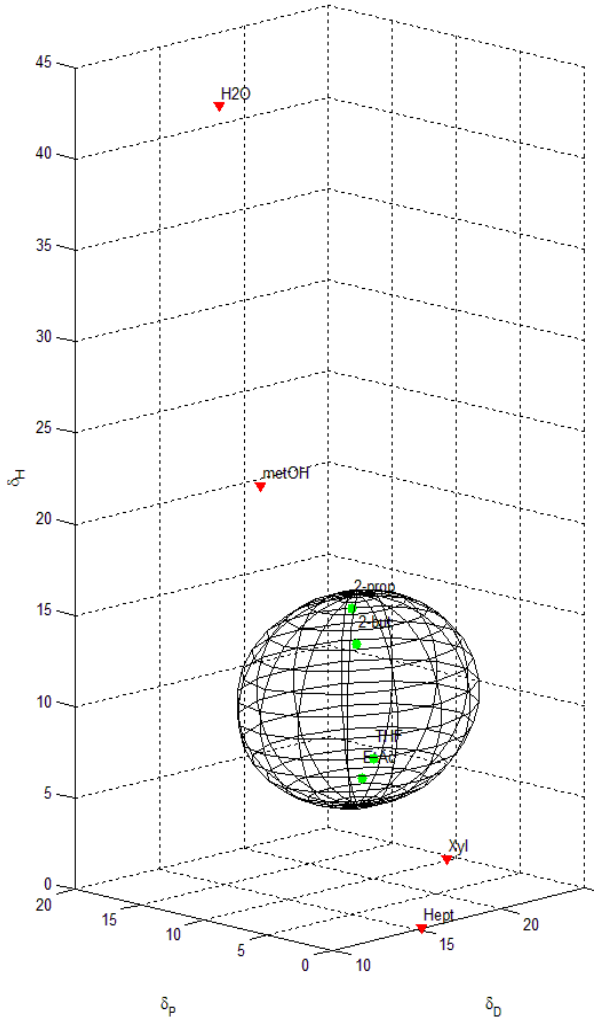


↓

The sphere center coordinates correspond to the three unknown HSP of the solute

* Fitting sphere program adapted from *J. Polym. Sci. Part B Polym. Phys* **47**(21), 2091 (2009) and solved in Matlab environment using the Nelder-Mead simplex algorithm.

Hansen solubility sphere calculated for HSAG-DDcP



Calculated HSP ($MPa^{0.5}$)

δ_D 16.5

δ_P 6.6

δ_H 11.2

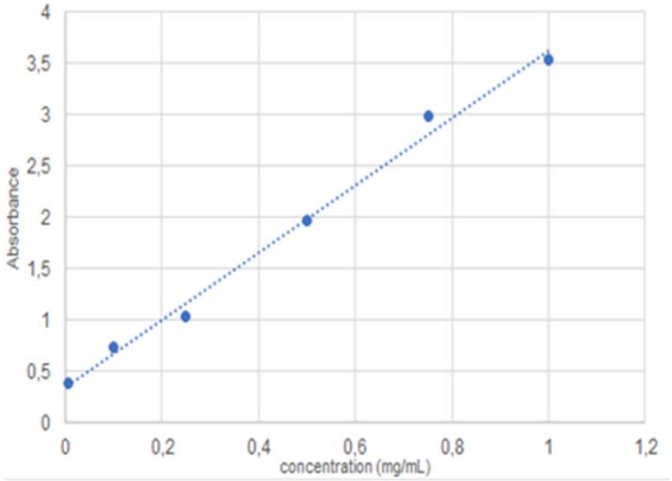
green circles: good solvents

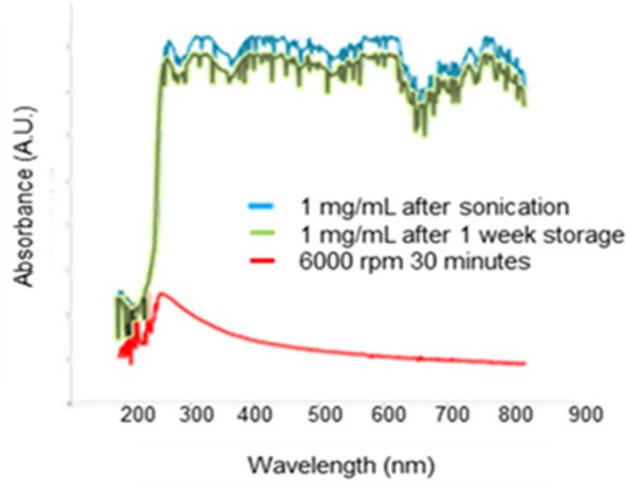
red triangles: bad solvents

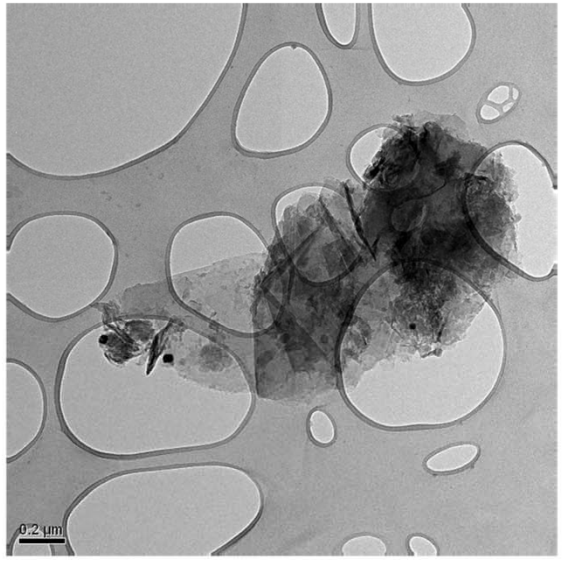
V. Barbera, A. Bernardi, A.Palazzolo, A. Rosengart, L. Brambilla, M. Galimberti Pure and Applied Chemistry

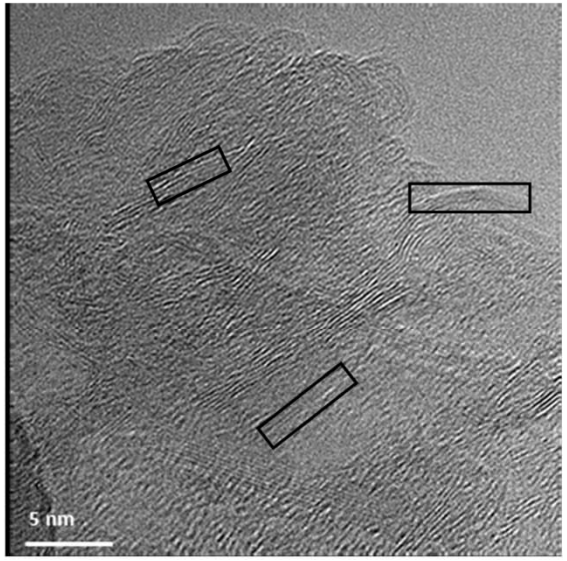
Ultimate dispersions and few layers graphene

By tuning the solubility parameter of graphene layers



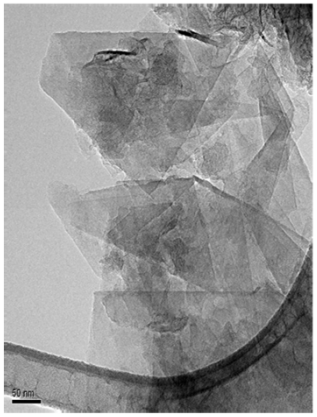






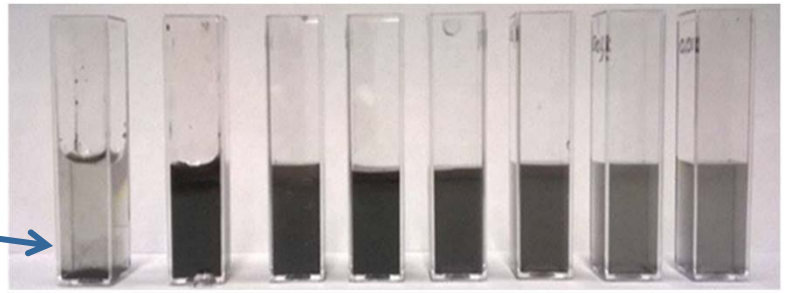
2-3 stacked layers

Graphene inks and varnishes



TEM micrograph

without NanoCarbon Up

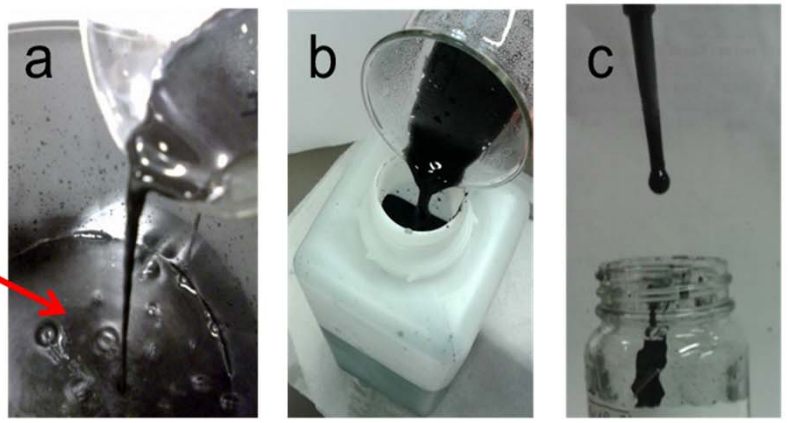


Conc (g/L): 10 5.0 2.5 1.0 0.50 0.25 0.10

freshly prepared

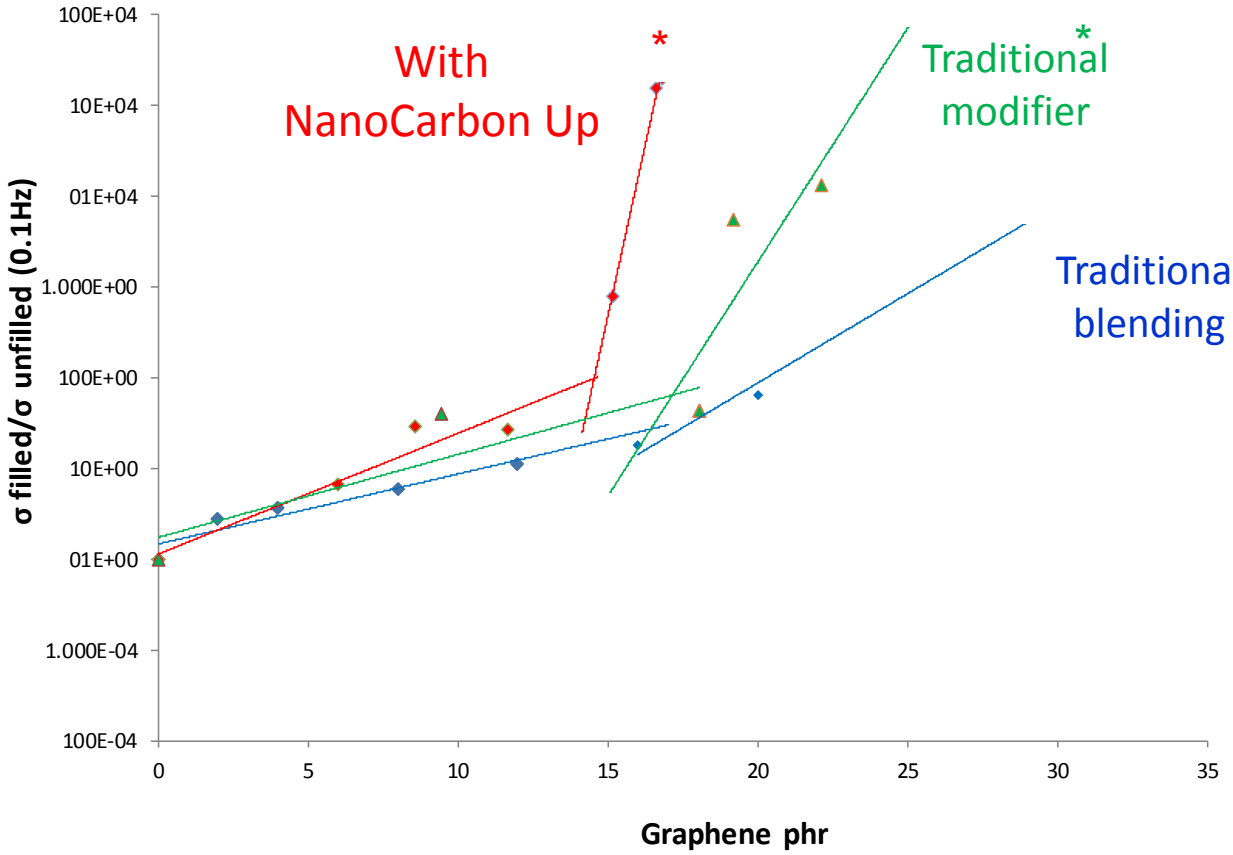
after 30 min centrifugation at 2000 rpm

Large scale preparation



Conc (g/L): 10 g/L, 30 g/L 200 g/L.

Graphene based polymer composites

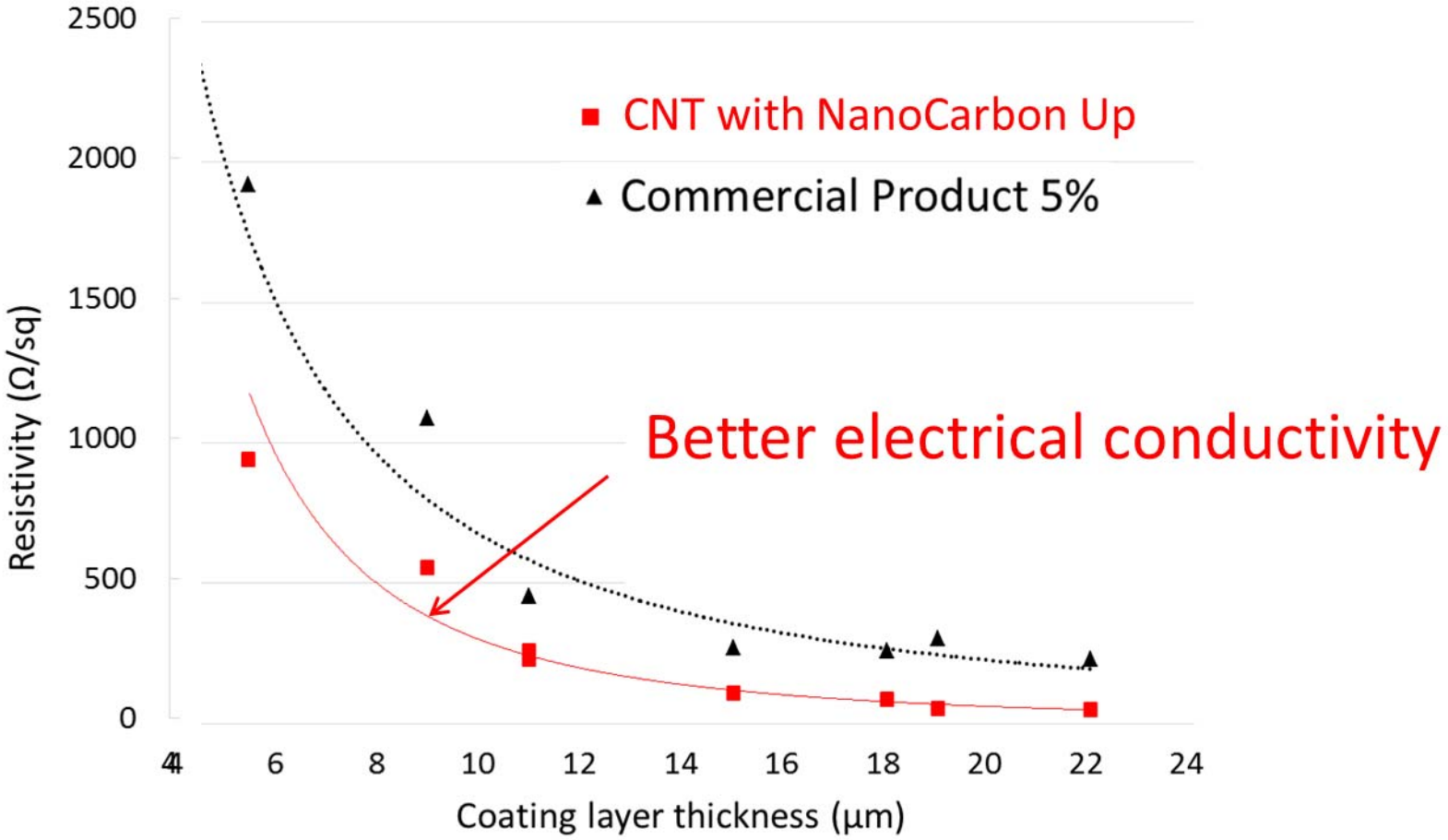


Polymer = NR

* From NR latex dispersion

☞ Better electrical conductivity

CNT inks and varnishes



Polyurethanes with better mechanical and barrier properties

2% by mass
in polyol

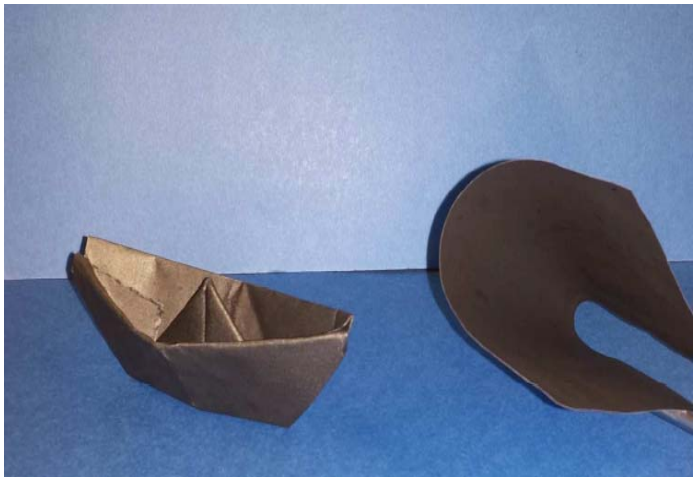
Large scale
preparation

Without
carbon material

With
carbon material

With
Nanocarbon Up

Graphene based bio-nanocomposites



Carbon papers

Monolithic aerogel

☞ Mechanical properties, electrical conductivity

NaoCarbon Up Technology

V. Barbera, A. Citterio, M. Galimberti, G. Leonardi, R. Sebastiano, S.U. Shisodia, A.M. Valerio.
WO/2015/189411 A1 (2015)

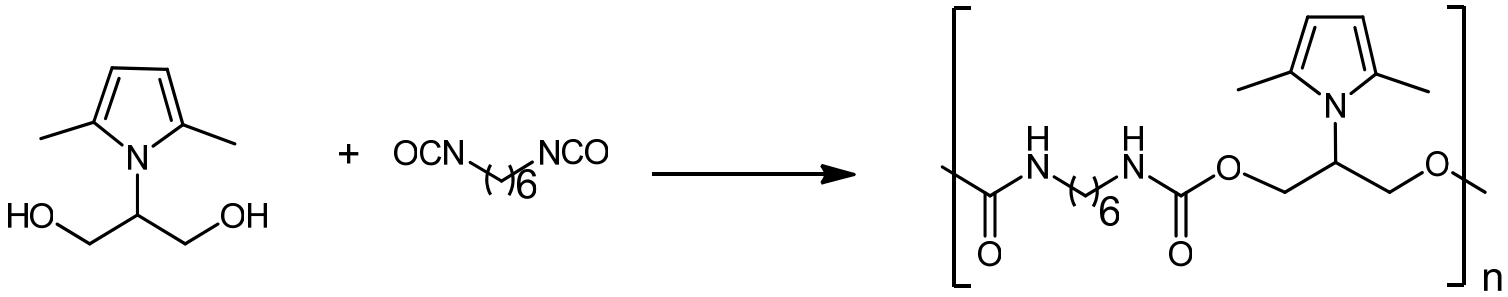
M. Galimberti, V. Barbera, R. Sebastiano, A. Citterio, G. Leonardi, A.M. Valerio.
WO/2016/050887 A1 (2016)

M. Galimberti, V. Barbera, R. Sebastiano, A. Truscello, A.M. Valerio.
WO/2016/023915 A1 (2016)

M. Galimberti, V. Barbera,
Italian Patent 102016000113012 (2016)

M. Galimberti, V. Barbera,
Italian Patent 102016000113070 (2016)

Polyurethane from SP and hexamethylenediisocyanate (HMMDI)



Temperature: 90°C

time (min): 60 – 120

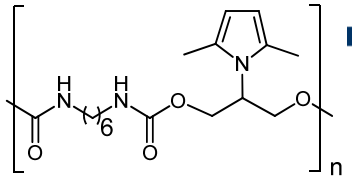
Without solvent

Mn (g/mol) = 1450 - 2400

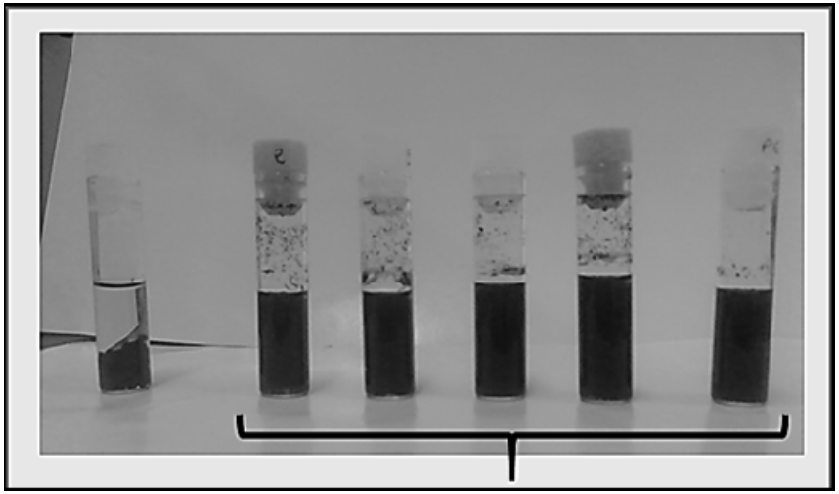
Polymer, (2015) 63 62–70

WO 2016 023915 A1

Polyurethane from SP and HMMDI



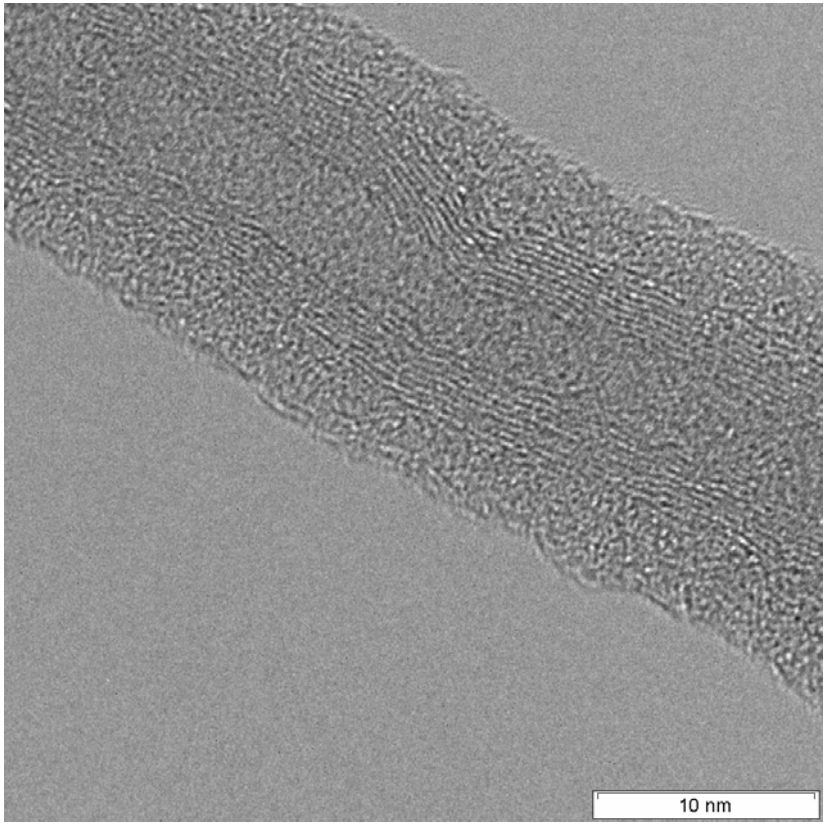
Ethyl acetate suspensions



CNT **CNT-PU/SP adducts**

Concentration (mg/mL): 0.5; 1; 1.5; 2.5; 5

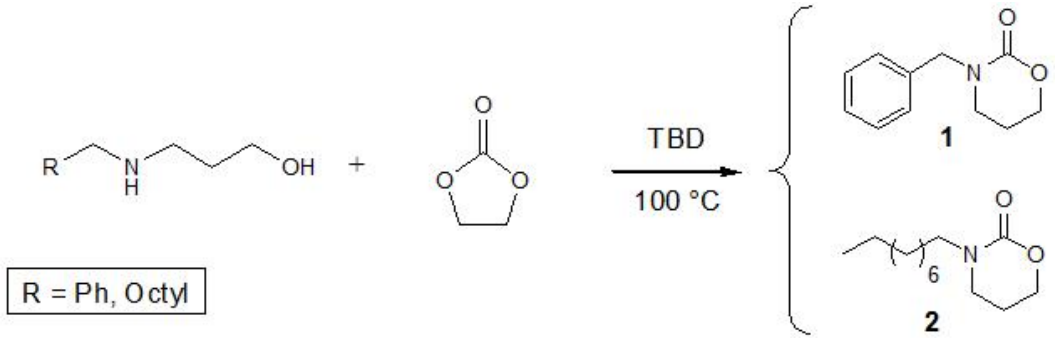
HRTEM micrograph of the PU/CNT adduct



Polymer, (2015) 63 62–70
WO 2016 023915 A1

Synthesis of 1,3-oxazin-2-ones 1-4 via DAC chemistry

Substituted 1,3-amino-alcohols + ethylene carbonate + a bicyclic nitrogen base



100% yield!

Base: 1,5,7-triazabicyclo[4.4.0]dec-5-ene (TBD)

☞ ☺

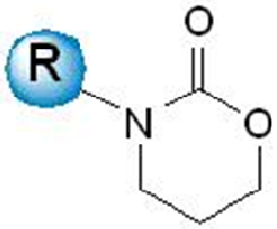
Atom efficiency: 100%

No toxic reagents

Easily available starting materials

Pretty simple reaction procedures

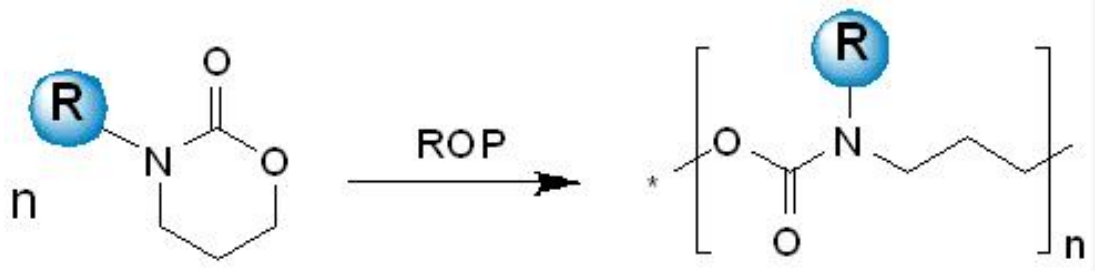
Minimal work up



Pure Appl. Chem. **2016**, *88* (3), 227–237.

F. Aricò, P. Tundo, University of Venice

Long lived ring opening polymerizations to poly(trimethylene urethanes)



R = octyl, benzyl, phenylamino

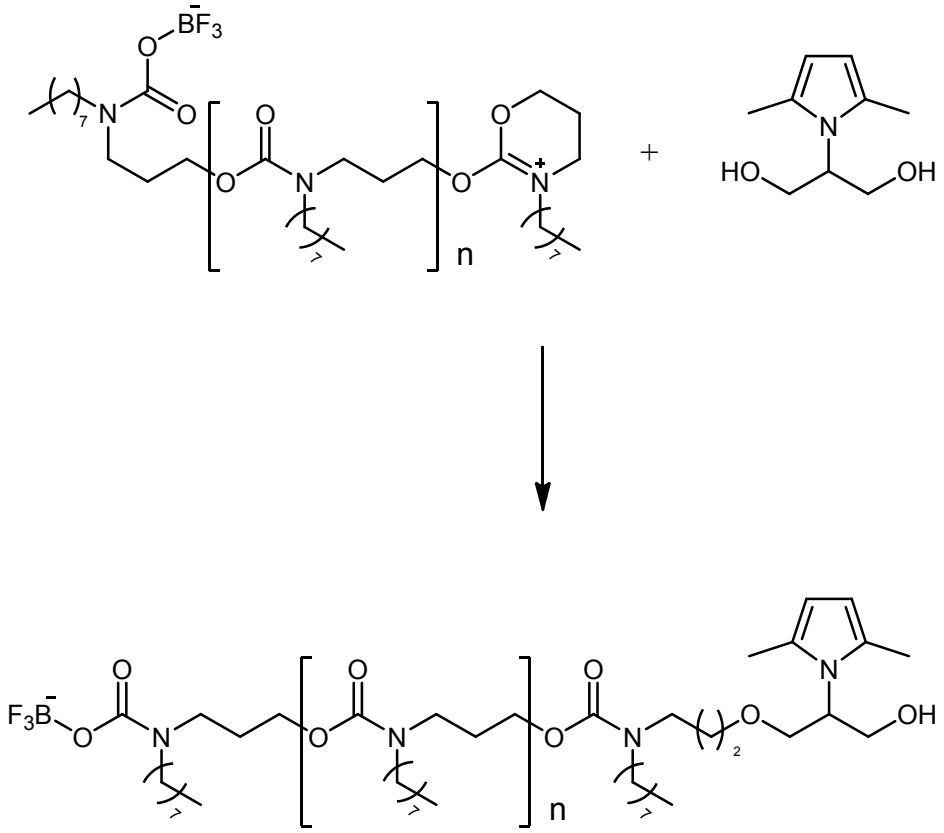
Cat: $BF_3O(Et)_2$ Monomer / Cat = 1 / 0.015

Temperature: 25 – 110°C

Time 12 hours

Without solvent

Polyurethanes from cationic ROP of 6-membered cyclic carbamates + SP



Adducts of PU and PU-SP with Carbon Nanotubes (CNT)

CNT + Acetone + PU → Sonication 15’

1 mg/mL

PU/CNT = 0.10 mass

CNT + Acetone + PU-SP → Sonication 15’

1 mg/mL

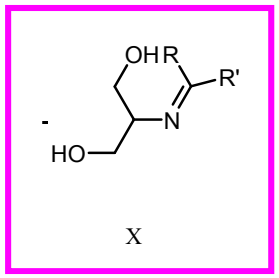
PU/CNT = 0.10 mass

Sulphur based crosslinking of rubbers

Reaction of serinol with carbonyl compounds.

With steric hindrance

and aromatic substituents

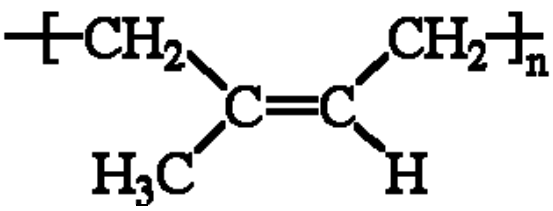


X

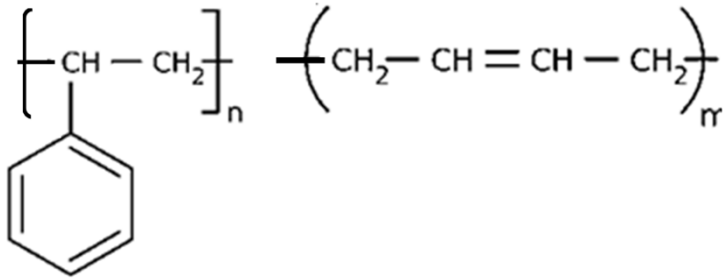
Imines

M. Galimberti, R. Sebastiano, V. Barbera, V. Cipolletti, G. Leonardi, S. Sun, L. Rossiello WO 2017/115253 A1

Rubbers

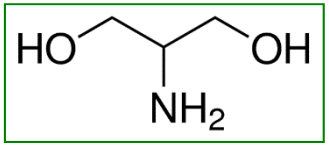


Poly(1,4-cis-isoprene)

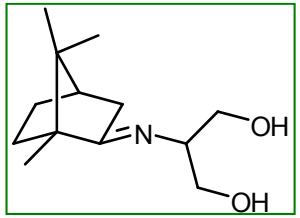


Poly(styrene-co-butadiene)

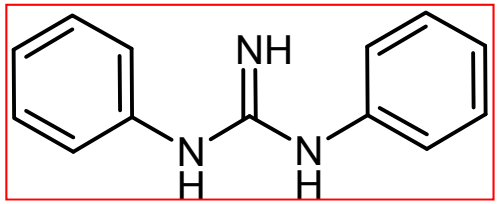
Serinol and serinol derivatives for rubber compounds



Serinol



Serinol camphor imine

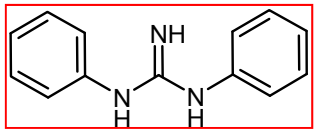
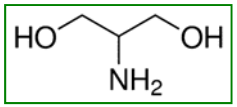
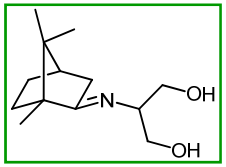


DPG

M. Galimberti, R. Sebastiano, V. Barbera, V. Cipolletti, G. Leonardi, S. Sun, L. Rossiello WO 2017/115253 A1

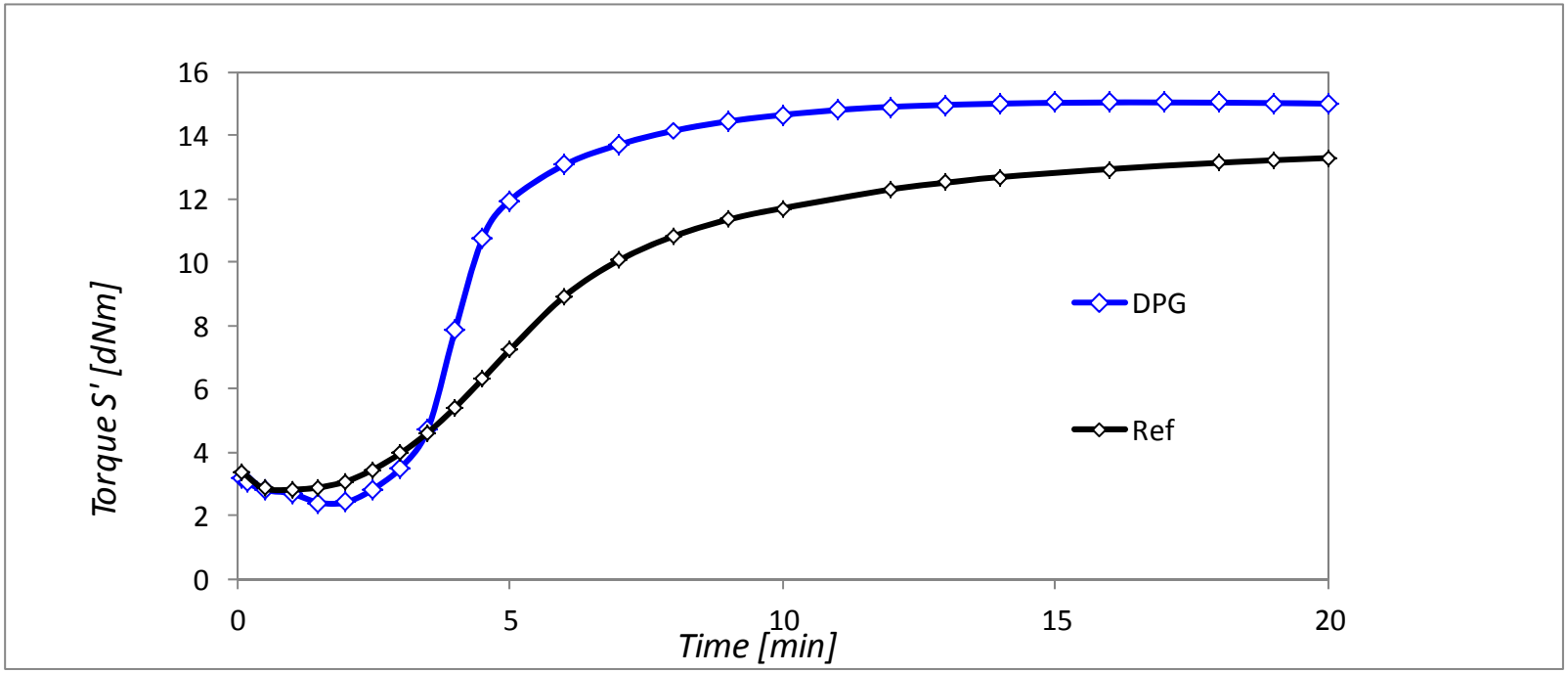
Serinol and serinol derivatives as secondary accelerators in silica based compounds

Ingredient	phr
S-SBR	96.3
NR	15
BR	15
Silica	65
Silane TESPT Si69	5.6
Oil MES	10
Stearic Acid	2
ZnO	2.5
6PPD	2
Sulphur	1.8
TBBS	1.2
Secondary accelerator	X

Secondary accelerator	X phr
	2.4
or	
	0.83
or	
	2.04

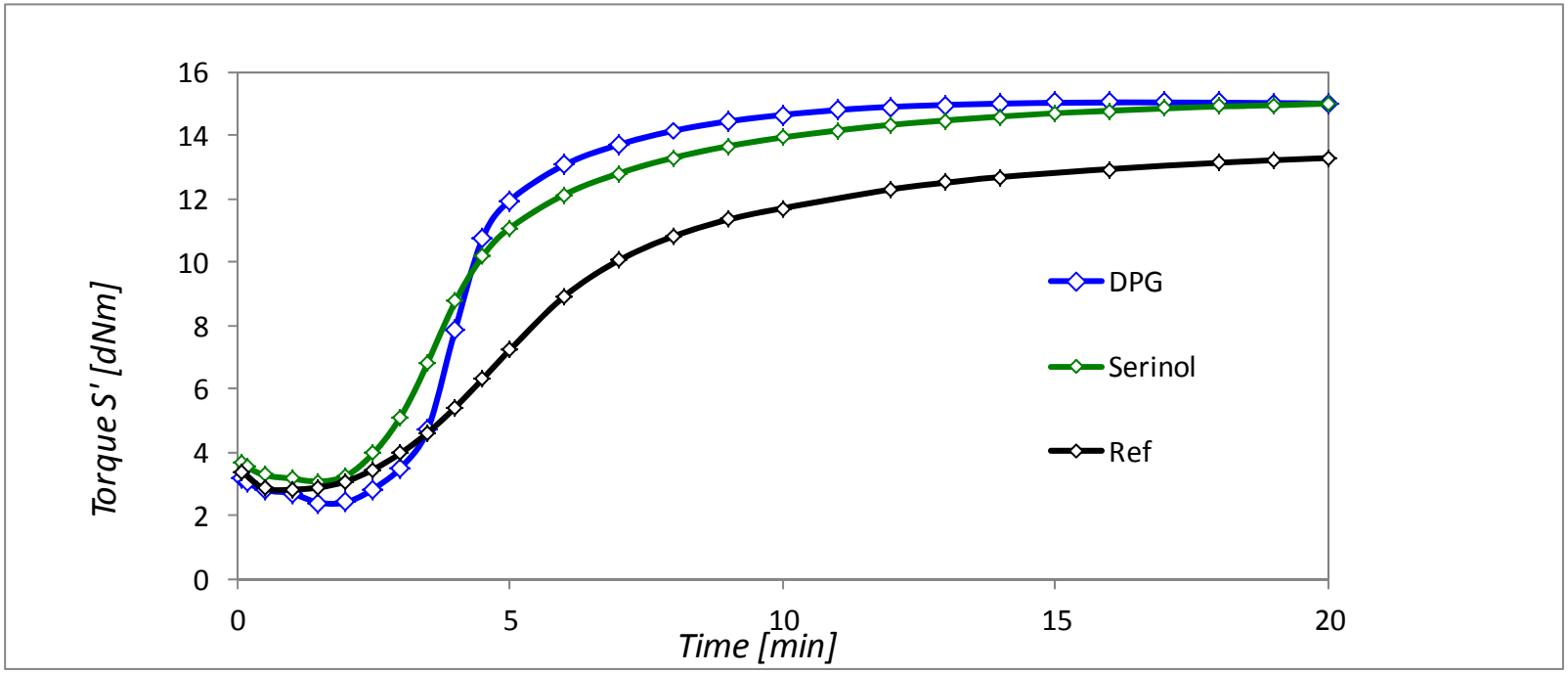
Secondary accelerators were used in equal molar amount

Curing at 170°C



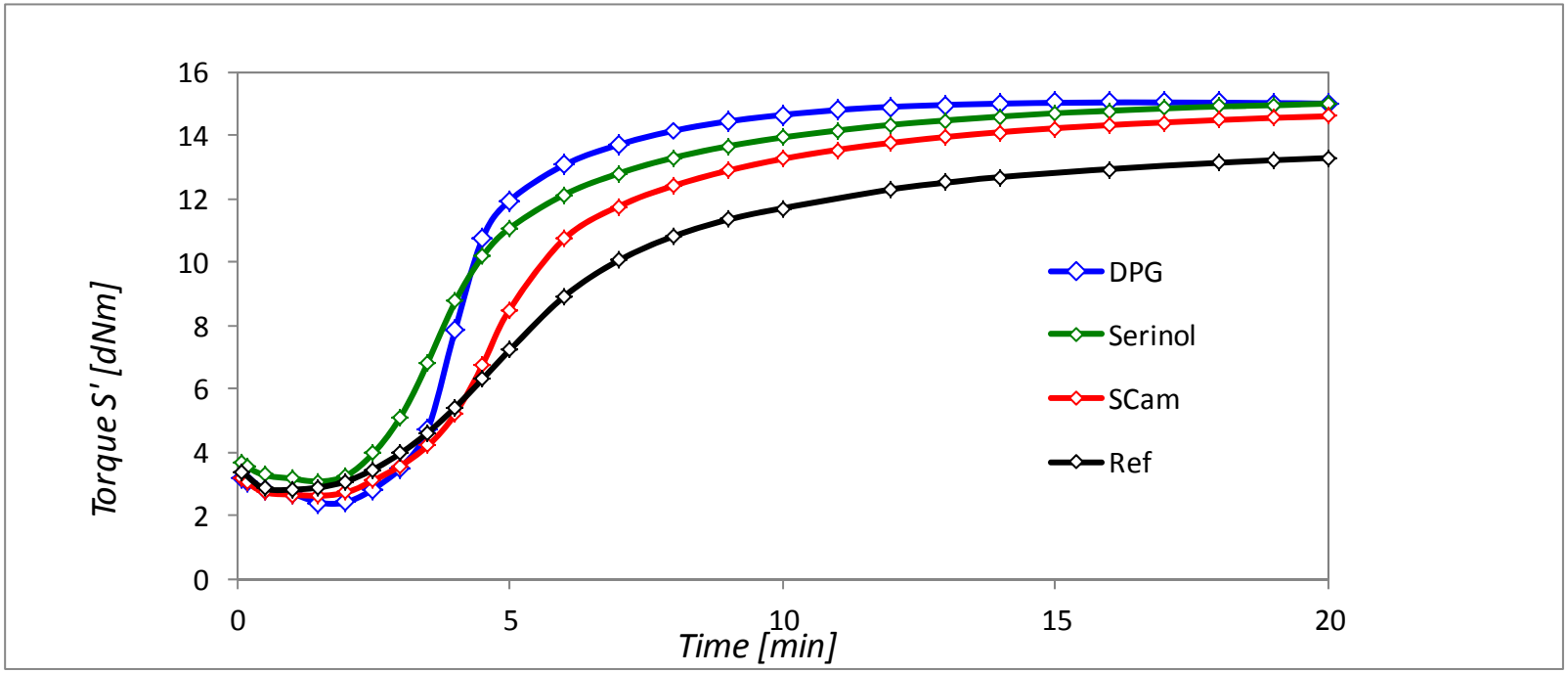
Secondary accelerator =			DPG
Parameter			
M_L	[dNm]	2.8	2.3
M_H	[dNm]	13.3	15.1
t_{s1}	[min]	2.8	2.9
t_{90}	[min]	11.5	7.1

Curing at 170°C



Secondary accelerator =			DPG	Serinol
Parameter				
M_L	[dNm]	2.8	2.3	3.1
M_H	[dNm]	13.3	15.1	15.0
t_{s1}	[min]	2.8	2.9	2.6
t_{90}	[min]	11.5	7.1	9.5

Curing at 170°C



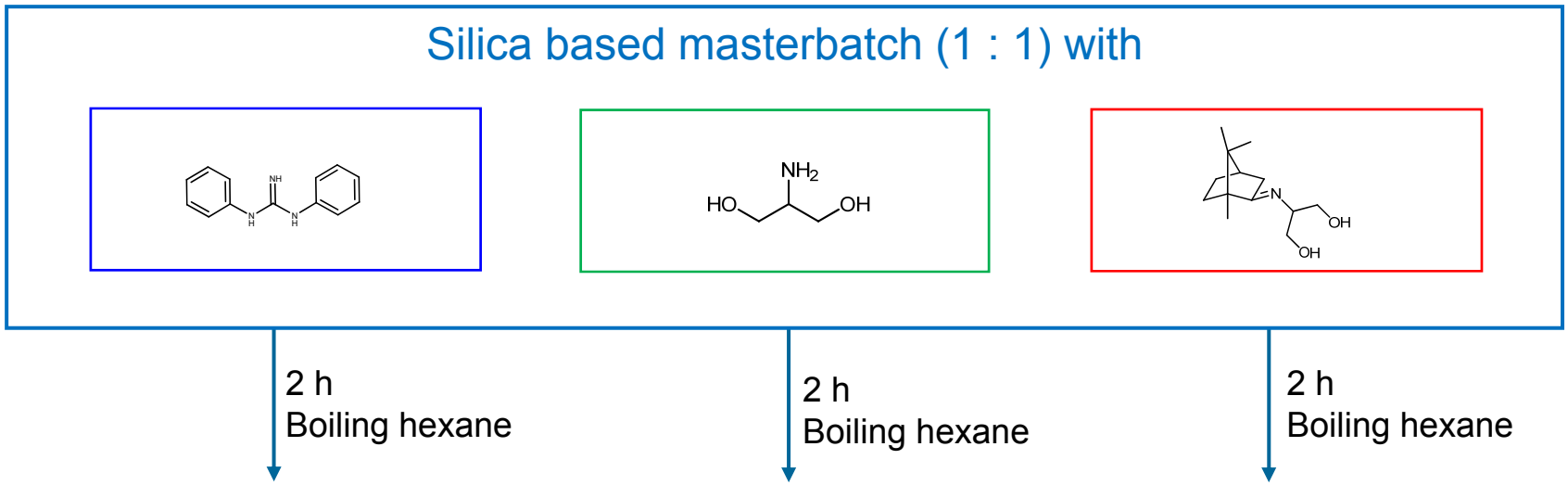
Secondary accelerator	=	DPG	Serinol	SCam
Parameter				
M_L [dNm]	2.8	2.3	3.1	2.6
M_H [dNm]	13.3	15.1	15.0	14.6
t_{s1} [min]	2.8	2.9	2.6	3.0
t_{90} [min]	11.5	7.1	9.5	10.5

Scorch time at 130°C

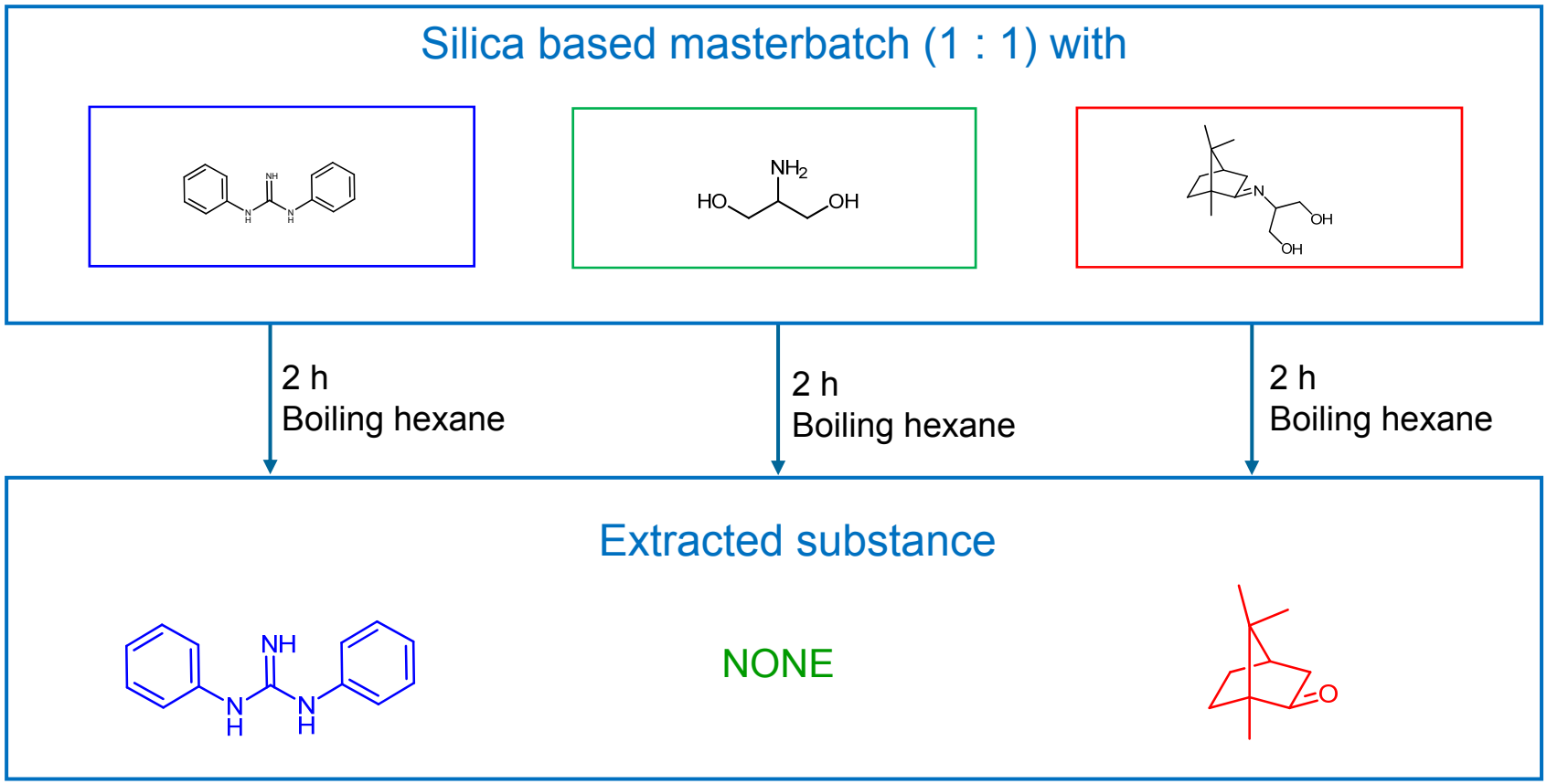
Secondary accelerator	=	DPG	Serinol	SCam
Parameter				
M_L dNm	7.1	6.1	7.0	6.5
t_{s5} [a] min	46.4	28.9	25.7	37.3

[a]Scorch time t_{s5}: = time needed to have an increase of torque of 5 dNm

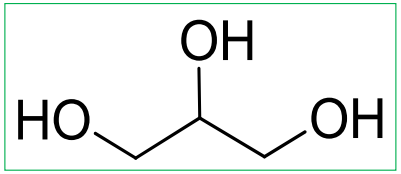
Solvent extraction of silica / secondary accelerator masterbatches

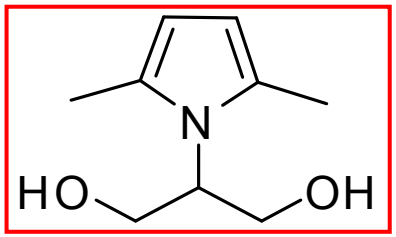


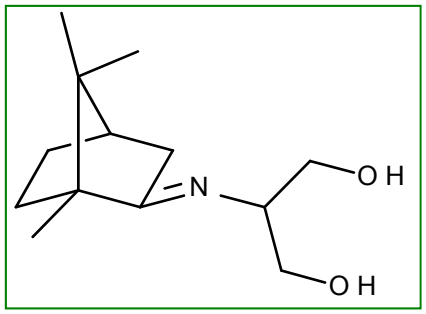
Solvent extraction of silica / secondary accelerator masterbatches

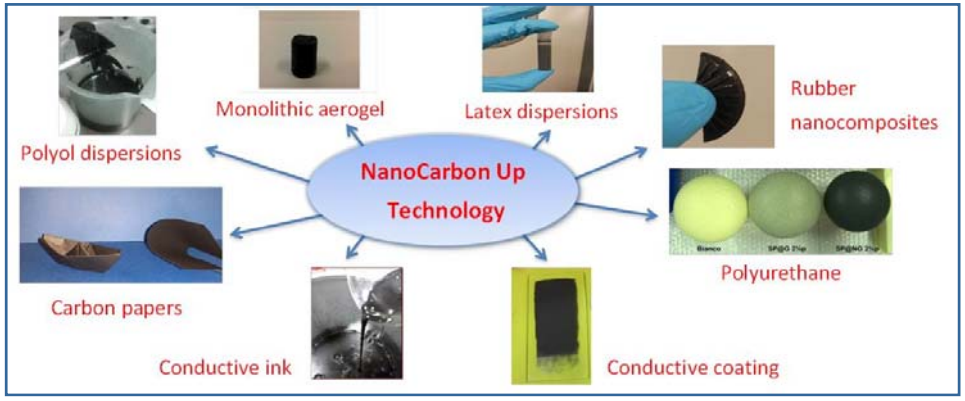


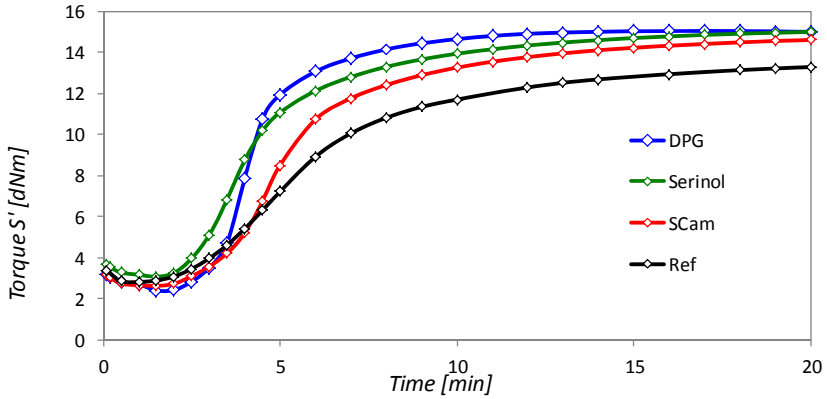
Conclusions











Thanks for the attention!

CPAC Rome Workshop 2018

March 19-21, 2018,
University of Washington Rome Center