Overview of the research areas:

ISCaMaP group

Materials preparation and binding to carbon allotropes
Catalysis ideas

M. Galimberti and V. Barbera

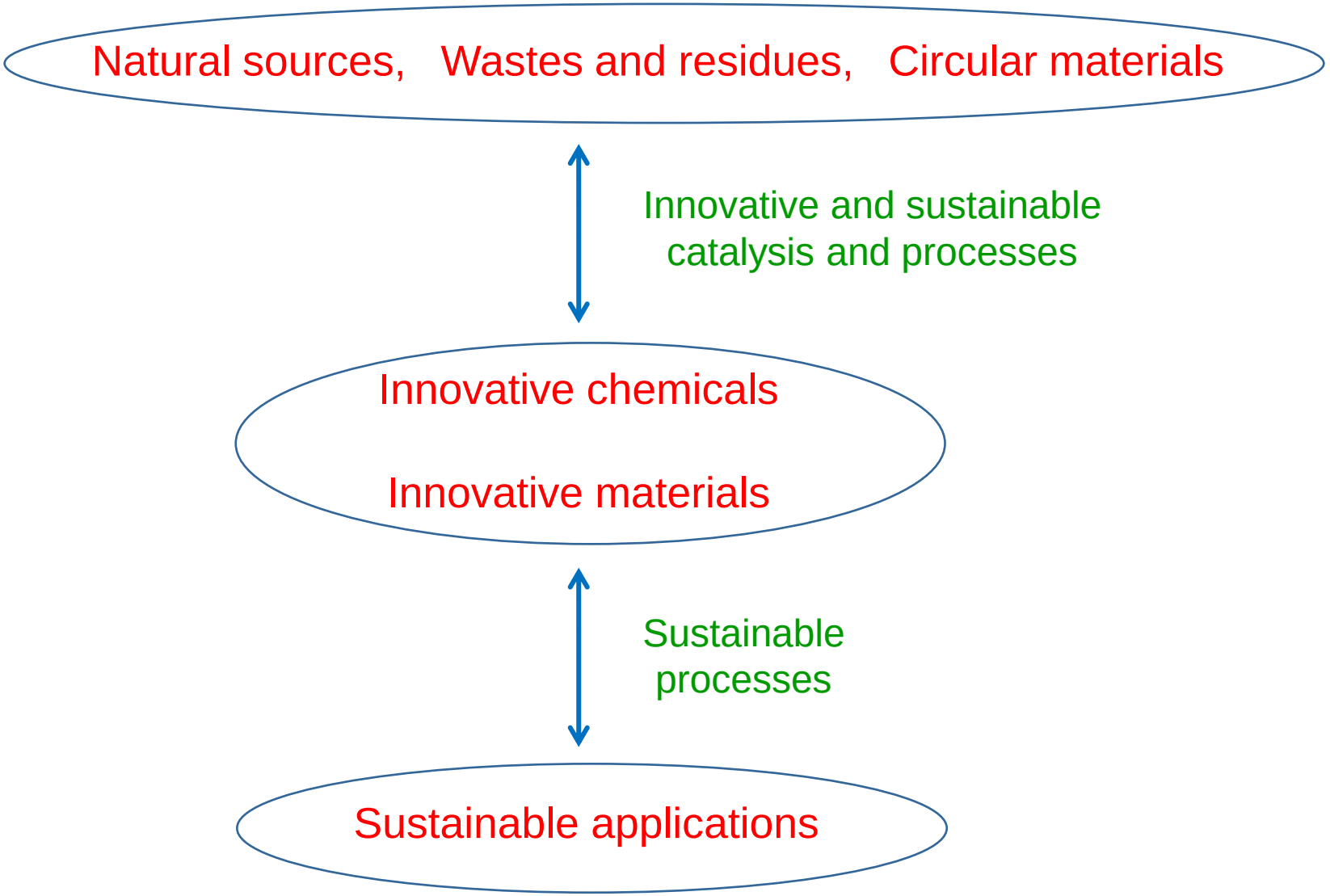
CPAC / Norse Biotech Project Workshop

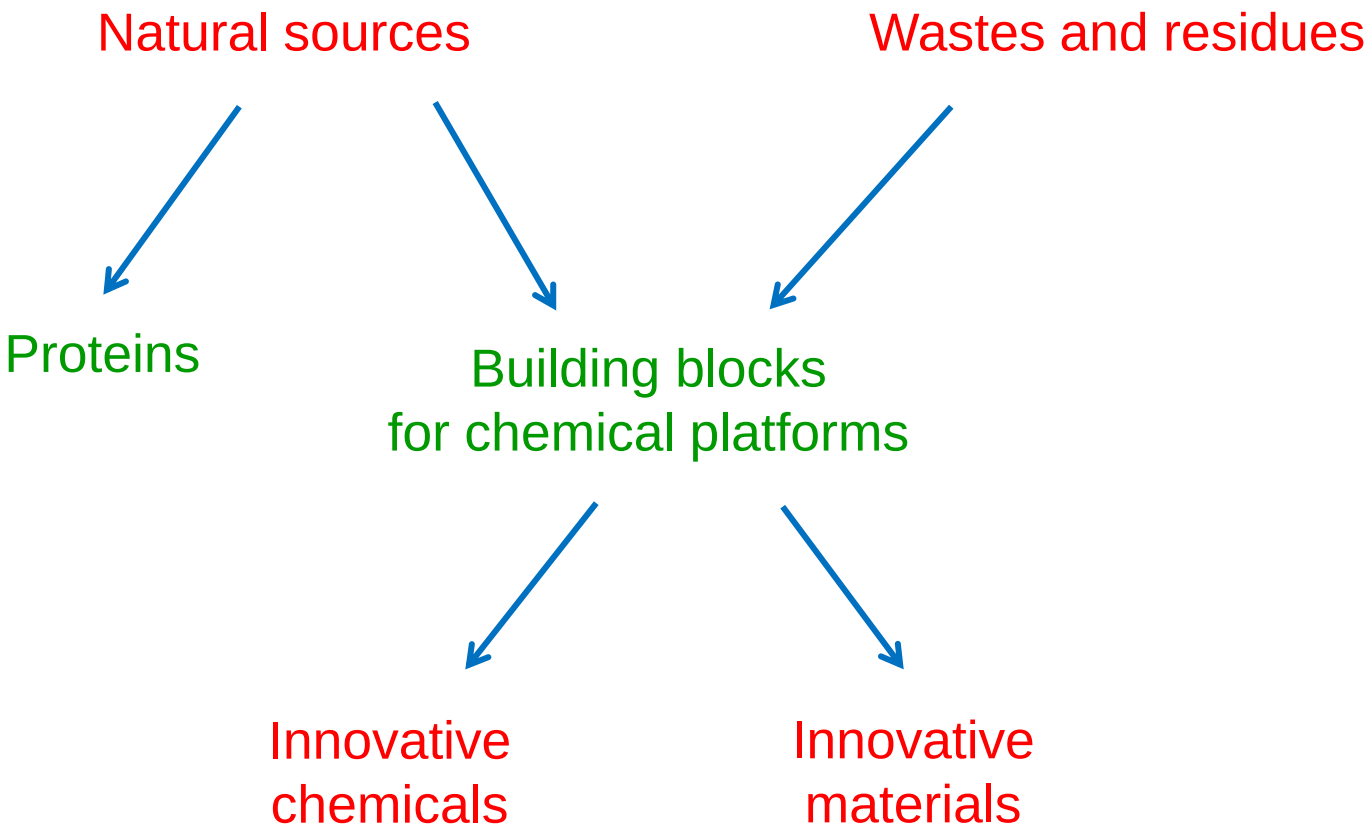
ISCaMaP

Innovative Sustainable Chemistry and Materials and Proteomics Group

Politecnico di Milano, Department of Chemistry, Materials and Chemical Engineering “G. Natta”

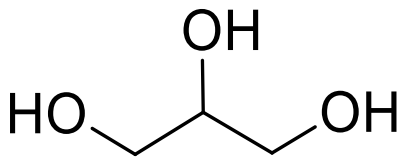
ISCaMaP Strategy: sustainability for innovation



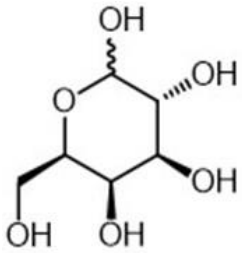


☞ Chemicals, Additives, Modifiers, Polymers

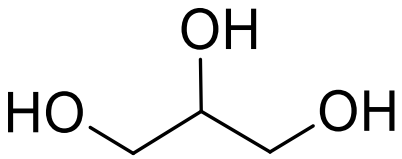
Innovative materials from C3 and C6 building blocks



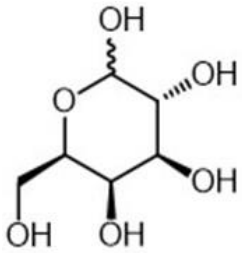
Glycerol, Sugars



Innovative materials from C3 and C6 building blocks



Glycerol, Sugars

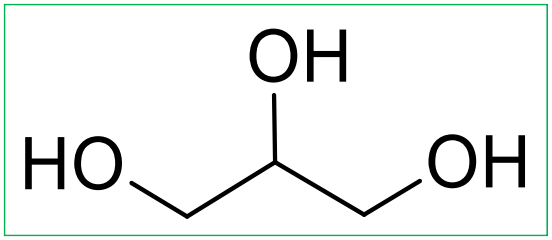


Biousourced substances + “Traditional Chemistry”

Innovative building blocks

C3 building blocks

Glycerol as the C3 building block



Propane-1,2,3-triol

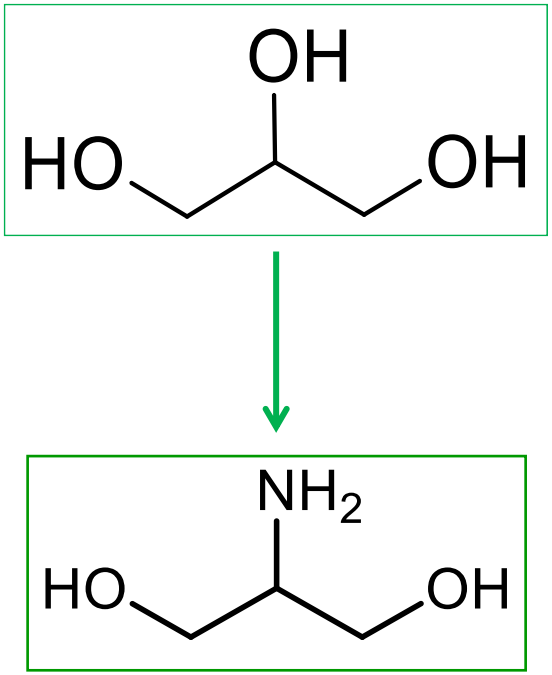
easily available, cheap raw material

main by-product of bio-diesel production

not toxic

biodegradable

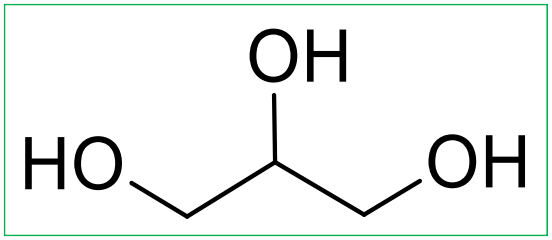
Glycerol as the C3 building block. From glycerol to serinol



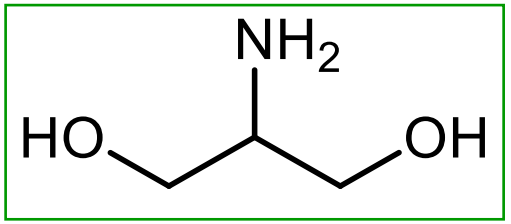
Propane-1,2,3-triol

2-Amino-1,3-propanediol

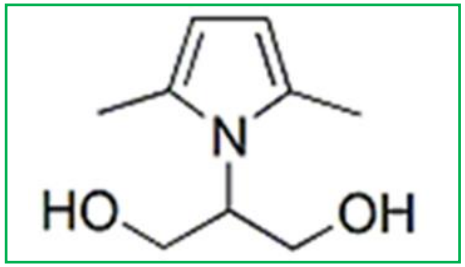
Glycerol as the C3 building block. From glycerol to serinol to serinol pyrrole



Propane-1,2,3-triol

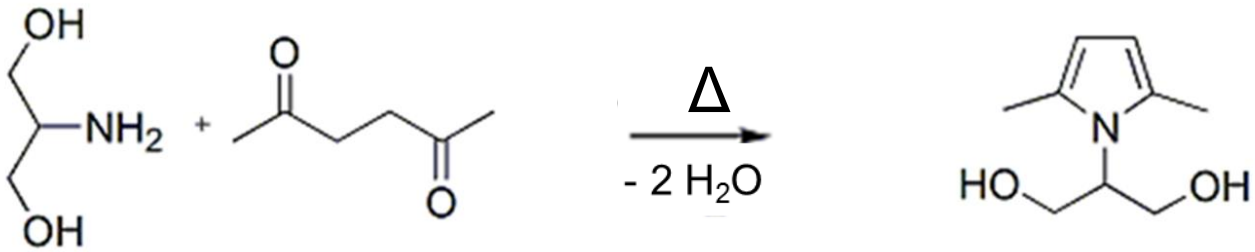


2-Amino-1,3-propanediol



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

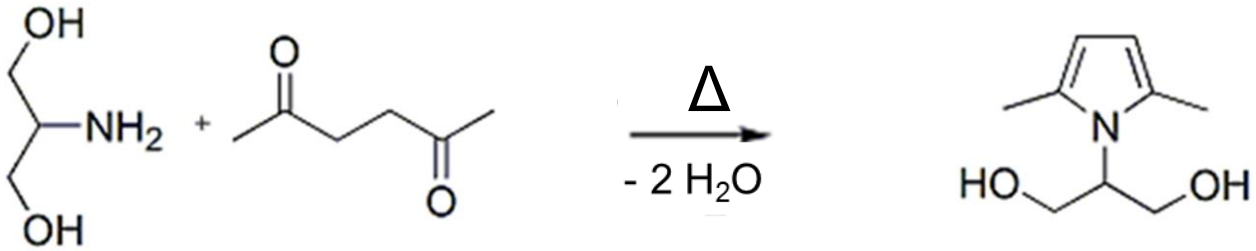
From serinol to serinol pyrrole

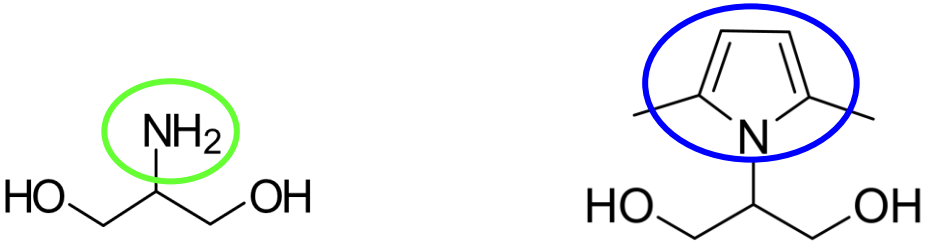


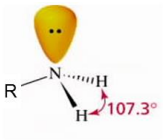
- Yield: at least 96%
- Atom efficiency: 85%
- Easy procedure
- No solvent
- By product: H_2O

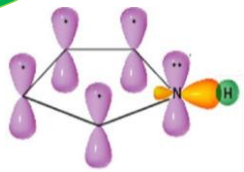
US10329253 B2; EP3154939 B1. V. Barbera, A. Citterio, M. Galimberti, G. Leonardi, R. Sebastiano, S. U. Shisodia, A. M. Valerio

From serinol to serinol pyrrole



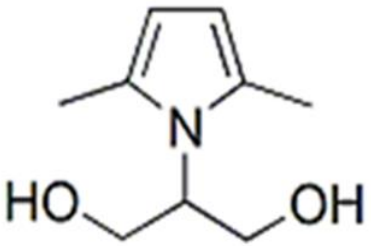






US10329253 B2; EP3154939 B1. V. Barbera, A. Citterio, M. Galimberti, G. Leonardi, R. Sebastiano, S. U. Shisodia, A. M. Valerio

Serinol pyrrole as a biosourced *Janus* molecule

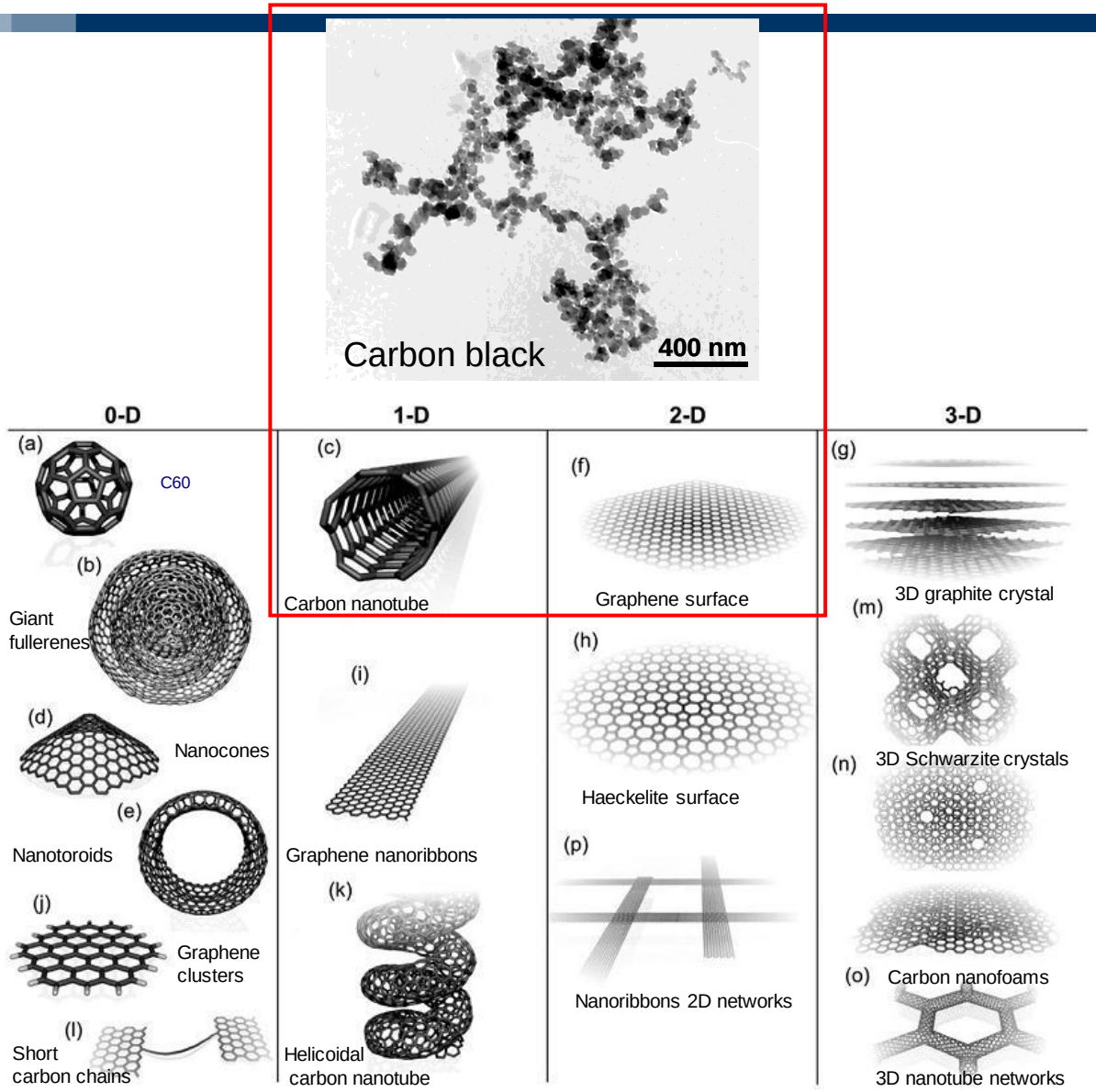


2-(2,5-dimethyl-1*H*-pyrrol-1-yl) -1,3-propanediol

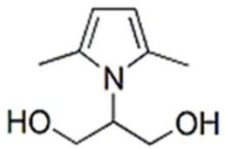
Serinol pyrrole - SP

Functionalization of sp^2 carbon allotropes

sp^2 Carbon allotropes (CA)



CA-SP Adducts - Preparation



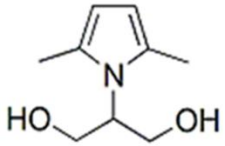
CA + SP

SP = 1 – 20 phc

phc = per hundred carbon

Galimberti, M., Barbera, V., Guerra, S., Conzatti, L., Castiglioni, C., Brambilla, L., A. Serafini, RSC Advances, 5(99), (2015) 81142-81152
Galimberti, M., Barbera, V., Sebastiano, R., Valerio A.M. Leonardi, G.,Citterio, US 2017 0275169 A1
Galimberti M., Barbera V., Guerra S., Bernardi A., Rubber Chemistry and Technology, 2017, 90(2), 285-307.

CA-SP Adducts - Preparation



Mechanical treatment

Ball Milling:

300 rpm, 6h

CA/SP-M

CA + SP

SP = 1 – 20 phc

phc = per hundred carbon

Galimberti, M., Barbera, V., Guerra, S., Conzatti, L., Castiglioni, C., Brambilla, L., A. Serafini, RSC Advances, 5(99), (2015) 81142-81152
Galimberti, M., Barbera, V., Sebastiano, R., Valerio A.M. Leonardi, G.,Citterio, US 2017 0275169 A1
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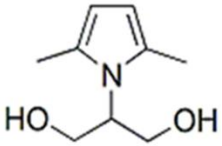


Mechanical treatment

Ball Milling:

300 rpm, 6h

CA/SP-M

CA + SP

SP = 1 – 20 phc

phc = per hundred carbon

80 - 180 C

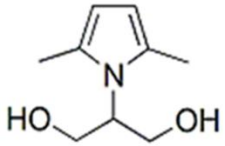
0,5 - 4 h

CA/SP-T

Thermal treatment

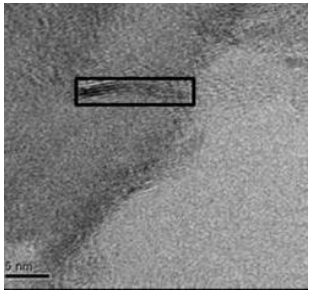
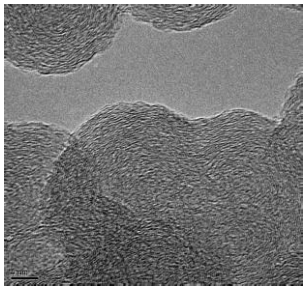
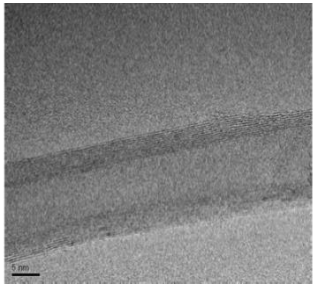
Galimberti, M., Barbera, V., Guerra, S., Conzatti, L., Castiglioni, C., Brambilla, L., A. Serafini, RSC Advances, 5(99), (2015) 81142-81152
Galimberti, M., Barbera, V., Sebastiano, R., Valerio A.M. Leonardi, G.,Citterio, US 2017 0275169 A1
Galimberti M., Barbera V., Guerra S., Bernardi A., Rubber Chemistry and Technology, 2017, 90(2), 285-307.

CA-SP Adducts - Yield of functionalization*



Thermal treatment

SP = 5 phc; 150°C, 2 h

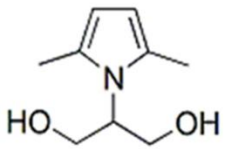
	HSAG	CB	MWCNT
			
BET Surface area: [m^2/g]	300	77	275
Functionalization Yield(%)*:	96	82	92

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

from TGA

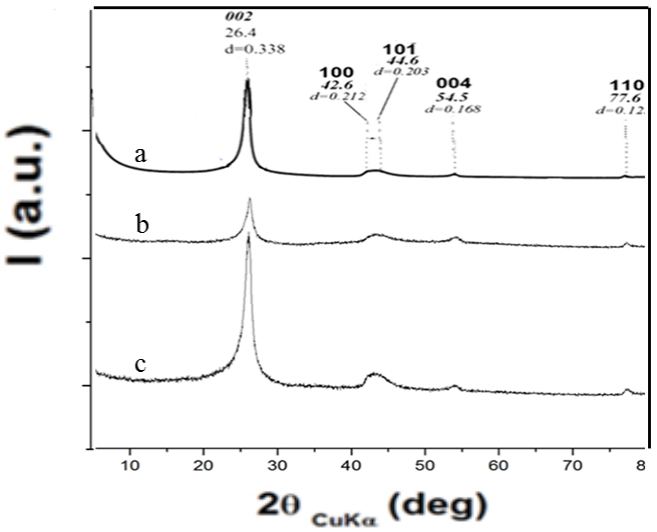
HSAG from Asbury, CB from Cabot, CNT from Nanocyl

Adducts of SP with high surface area graphite (HSAG)



CA/SP → Soxhlet extraction in acetone

WAXD

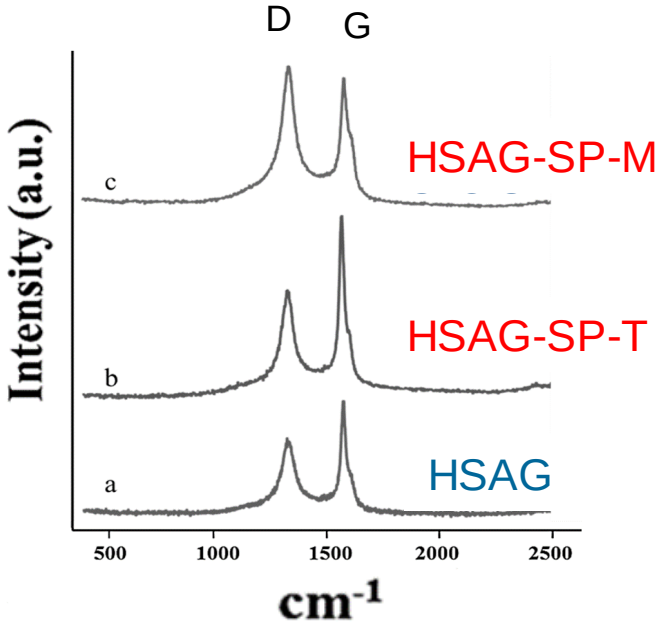


HSAG

HSAG-SP-M

HSAG-SP-T

Raman



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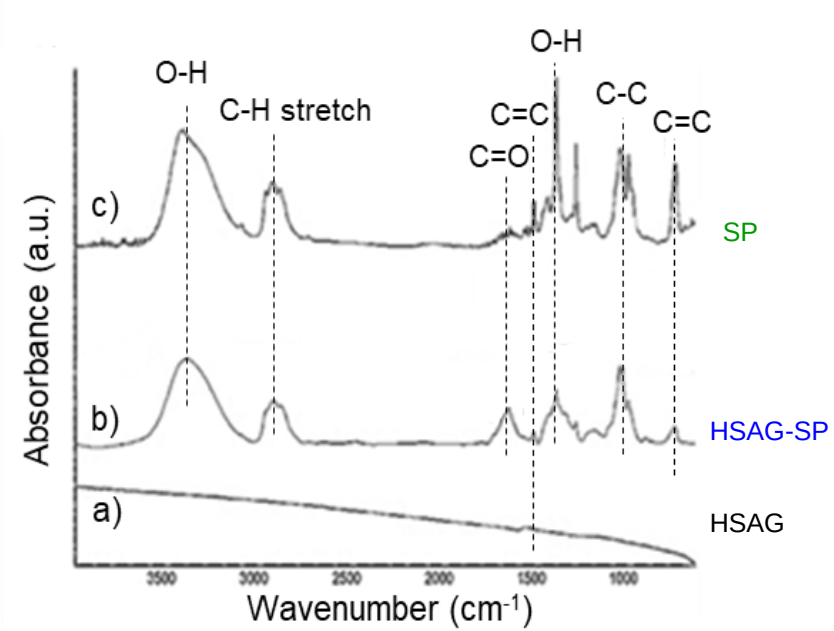
Galimberti M., Barbera V., Guerra S., Bernardi A., Rubber Chemistry and Technology, 2017, 90(2), 285-307.

Adducts of SP with high surface area graphite (HSAG)

CA/SP-T → Soxhlet extraction in acetone

FT-IR



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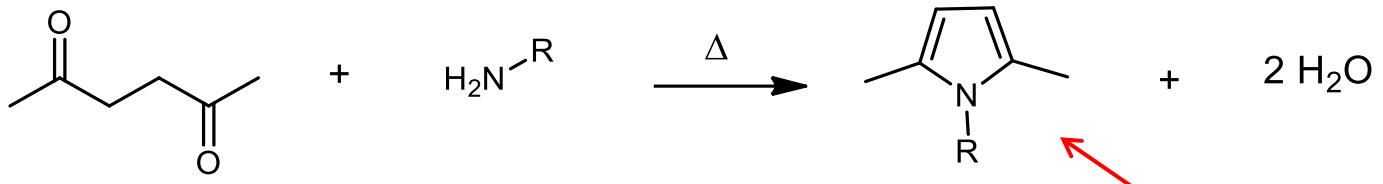
Galimberti M., Barbera V., Guerra S., Bernardi A., Rubber Chemistry and Technology, 2017, 90(2), 285-307.

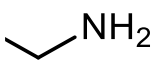
Pyrrole compounds (PyC) from neat Paal Knorr reaction



Same reaction conditions used for SP

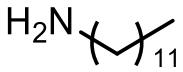
PyC

Yield %



80

75



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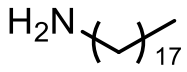

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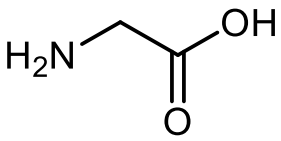

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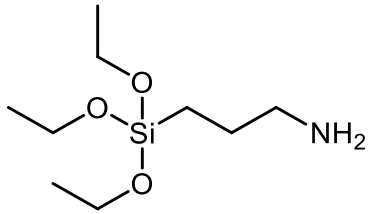
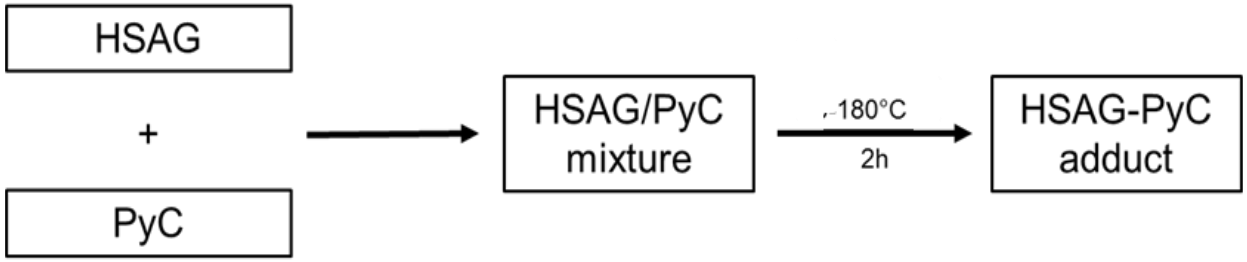


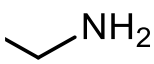
70

V. Barbera, A. Bernardi, A.Palazzolo, A. Rosengart, L. Brambilla, M. Galimberti Pure and Applied Chemistry 2018, 90(2), 253–270

HSAG / PyC adducts



Functionalization Yield %

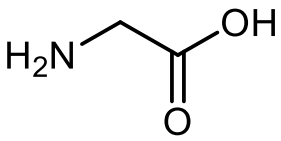


57

53

73

55



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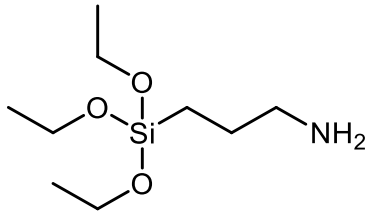
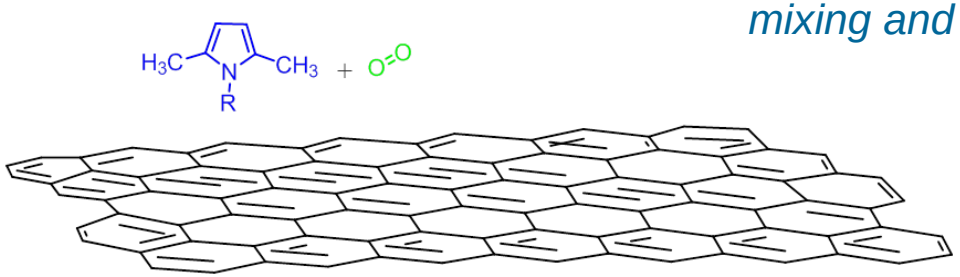


70

V. Barbera, A. Bernardi, A.Palazzolo, A. Rosengart, L. Brambilla, M. Galimberti Pure and Applied Chemistry 2018, 90(2), 253–270

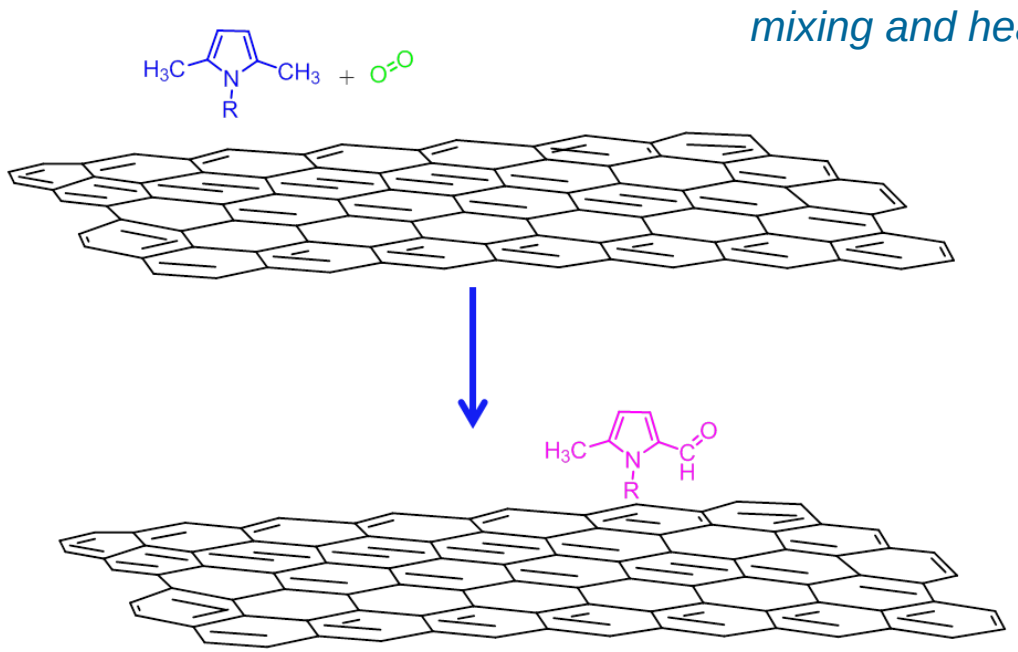
Mechanism for the formation of CA/PyC adducts

mixing and heating



V. Barbera, L. Brambilla, M. Milani, A. Palazzolo, C. Castiglioni, A. Vitale, R. Bongiovanni, M. Galimberti, 2019. Nanomaterials, 9(1), p.44.

Mechanism for the formation of CA/PyC adducts



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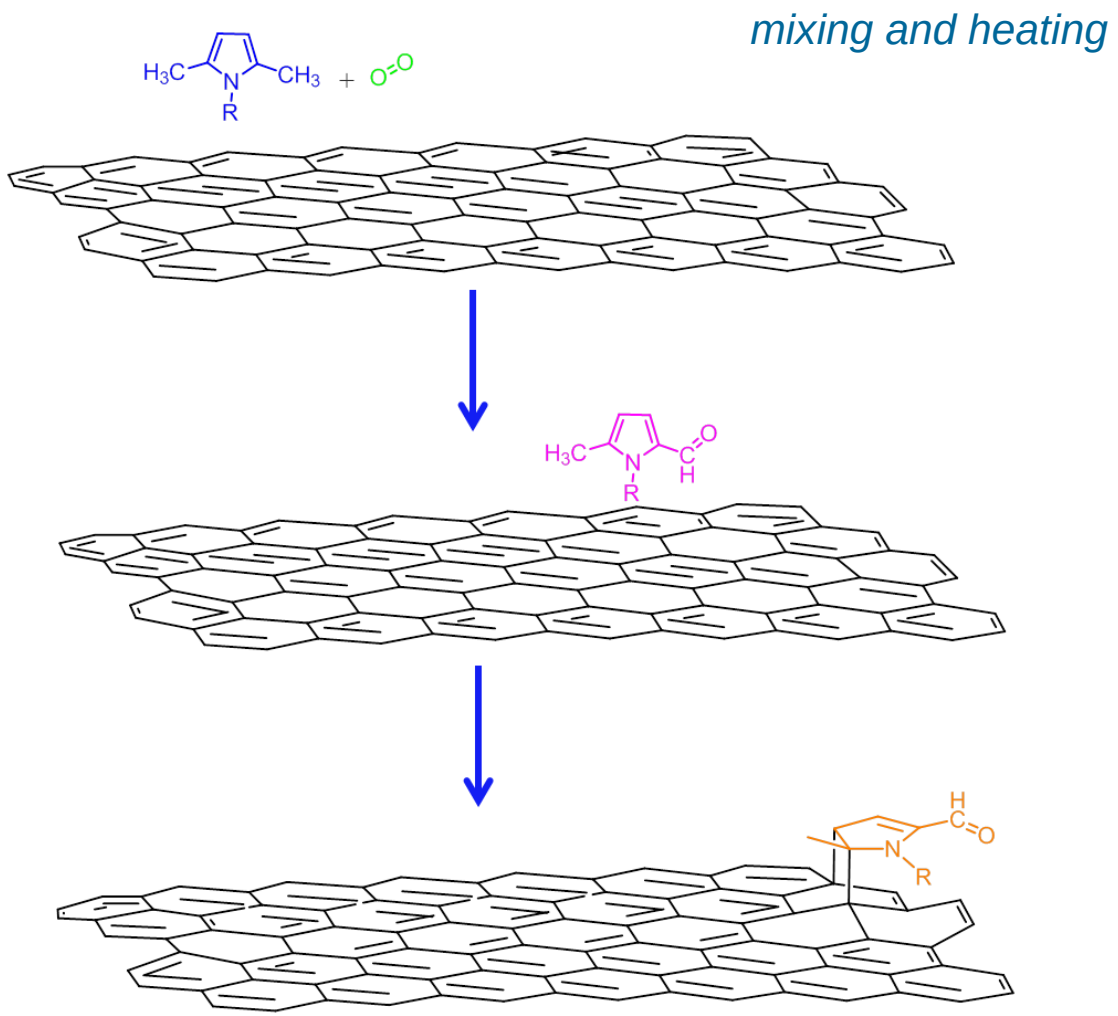
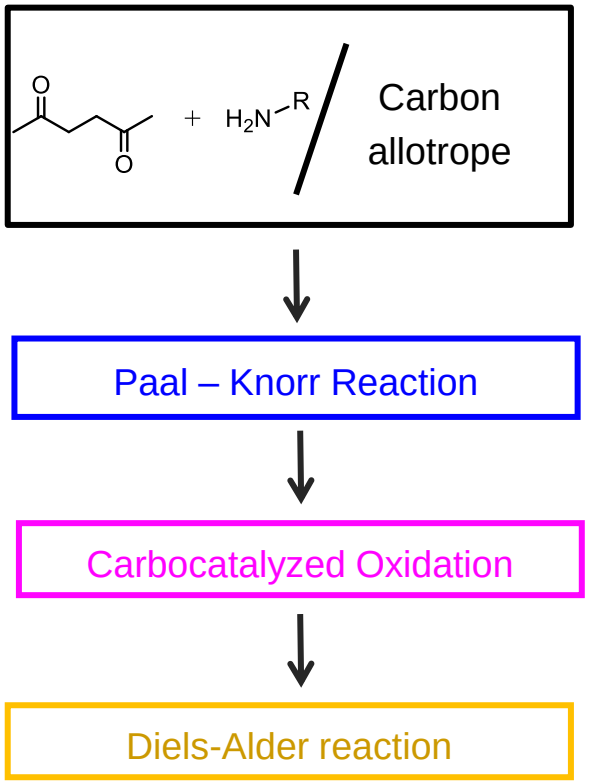
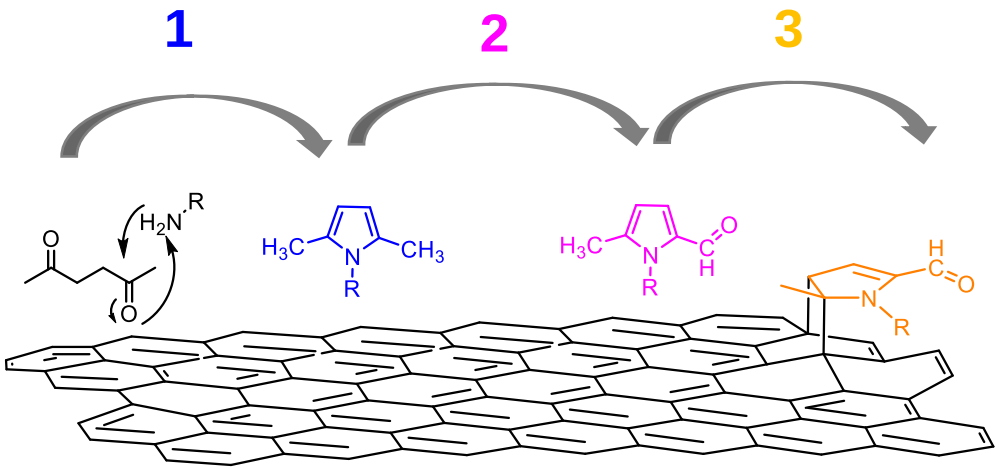


V. Barbera, L. Brambilla, M. Milani, A. Palazzolo, C. Castiglioni, A. Vitale, R. Bongiovanni, M. Galimberti, 2019. Nanomaterials, 9(1), p.44.

Mechanism for the formation of CA/PyC adducts

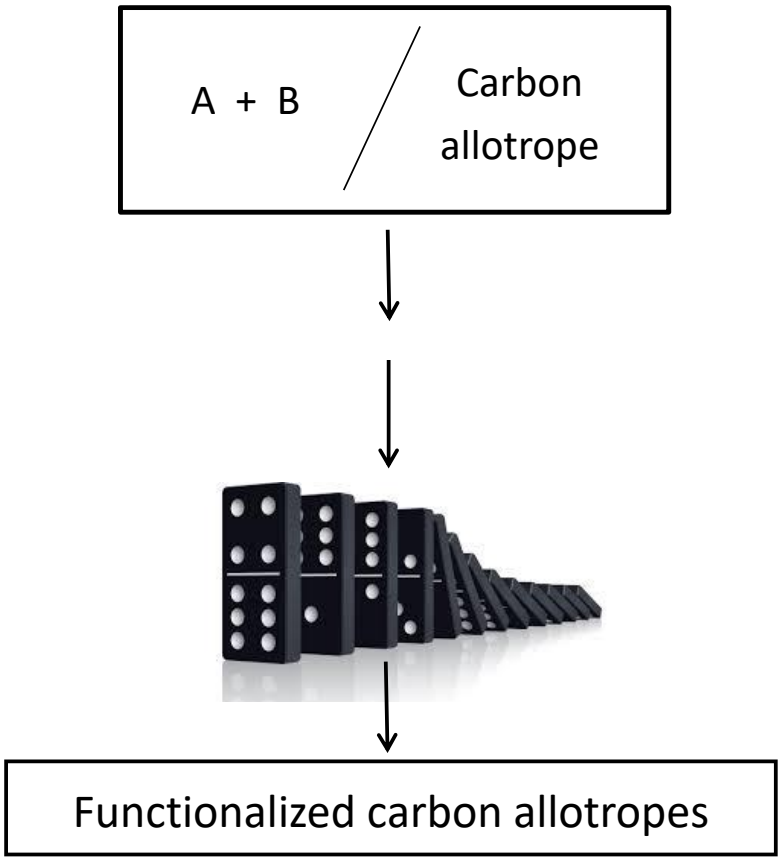
Domino reaction





V. Barbera, L. Brambilla, M. Milani, A. Palazzolo, C. Castiglioni, A. Vitale, R. Bongiovanni, M. Galimberti, 2019. Nanomaterials, 9(1), p.44.

The CA/PyC adducts



- Functional group:
  from few % to 20%
- Functionalization yield:
  from 85% to quantitative
- Covalent bond
  between functional group and carbon allotrope
- Bulk structure of graphitic materials:
  substantially unaltered

V. Barbera, A. Citterio, M. Galimberti, G. Leonardi, R. Sebastiano, S.U. Shisodia, A.M. Valerio. *US10329253B2*
M. Galimberti, V. Barbera, R. Sebastiano, A. Citterio, G. Leonardi, A.M. Valerio. *US10160652B2*
M. Galimberti, V. Barbera, R. Sebastiano, A. Truscello, A.M. Valerio. *EP3180379B1*
M. Galimberti, V. Barbera, *EP3538511A1*
M. Galimberti, V. Barbera, *EP3538481A1*

CA / PyC adducts - Tuning of solubility parameters

Experimental determination

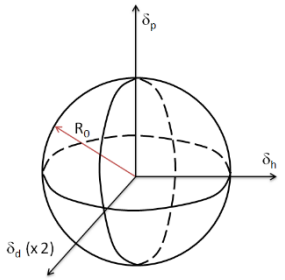
↓

Stable suspensions
in solvents
with different δ

Theoretical predictions

↓

Computational model:
Hansen solubility parameters



Evaluation of solubility parameters of HSAG-PyC - Experiments

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

No suspension: **bad**

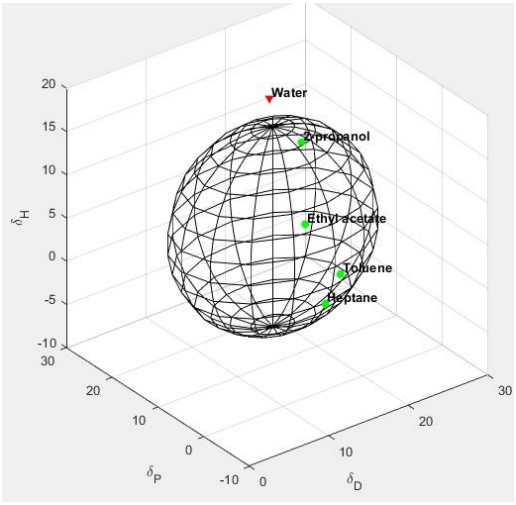
Unstable suspension: **bad**

Stable suspension: **good**

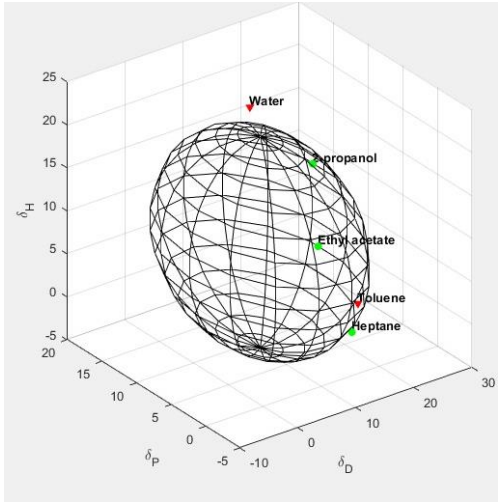
V. Barbera, A. Bernardi, A.Palazzolo, A. Rosengart, L. Brambilla, M. Galimberti Pure and Applied Chemistry 2018, 90(2), 253–270

Evaluation of solubility parameters of HSAG-PyC - Hansen sphere

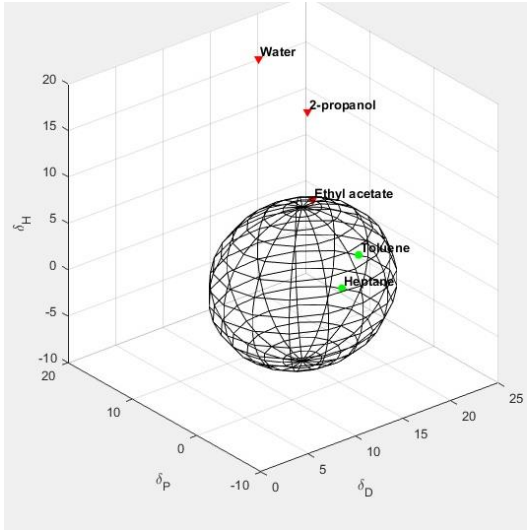
HSAG-TMP



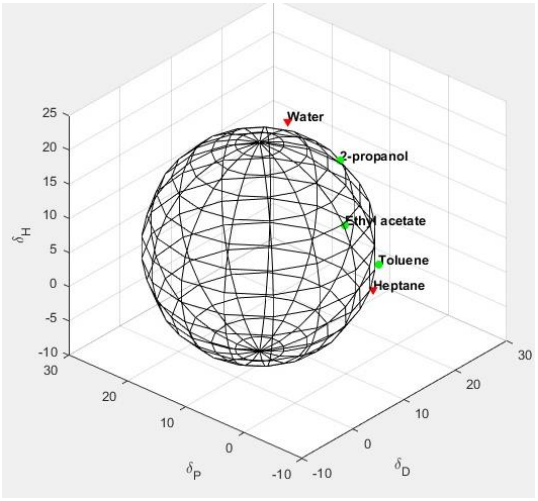
HSAG-DDcP



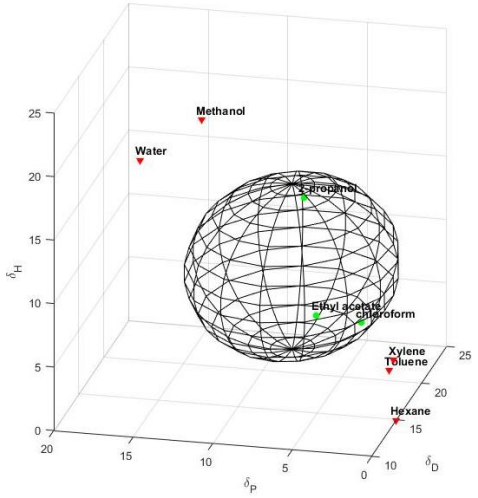
HSAG-APTESP

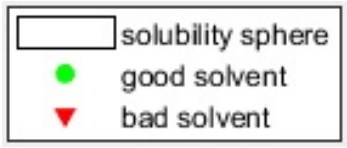


HSAG-GlyP



HSAG-SP





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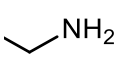
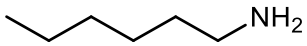
Evaluation of solubility parameters of HSAG-PyC - δ values

Sample	δ_D	δ_P	δ_H	Radius
HSAG	17.8	3.1	5.7	1.0
HSAG-TMP	14.6	10.3	5.6	11.6
HSAG-DDcP	8.5	7.5	8.3	12.3
HSAG-APTESP	12.7	2.3	0.5	8.3
HSAG-SP	12.8	2.0	8.9	13.8
HSAG-GlyP	6.9	12.1	5.3	15.3

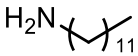
Amount of PyC on HSAG: about 5% mol



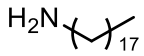
TMP



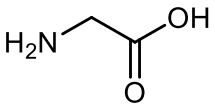
EP



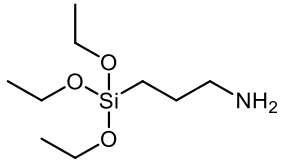
DDcP



ODcP



GlyP



APTESP

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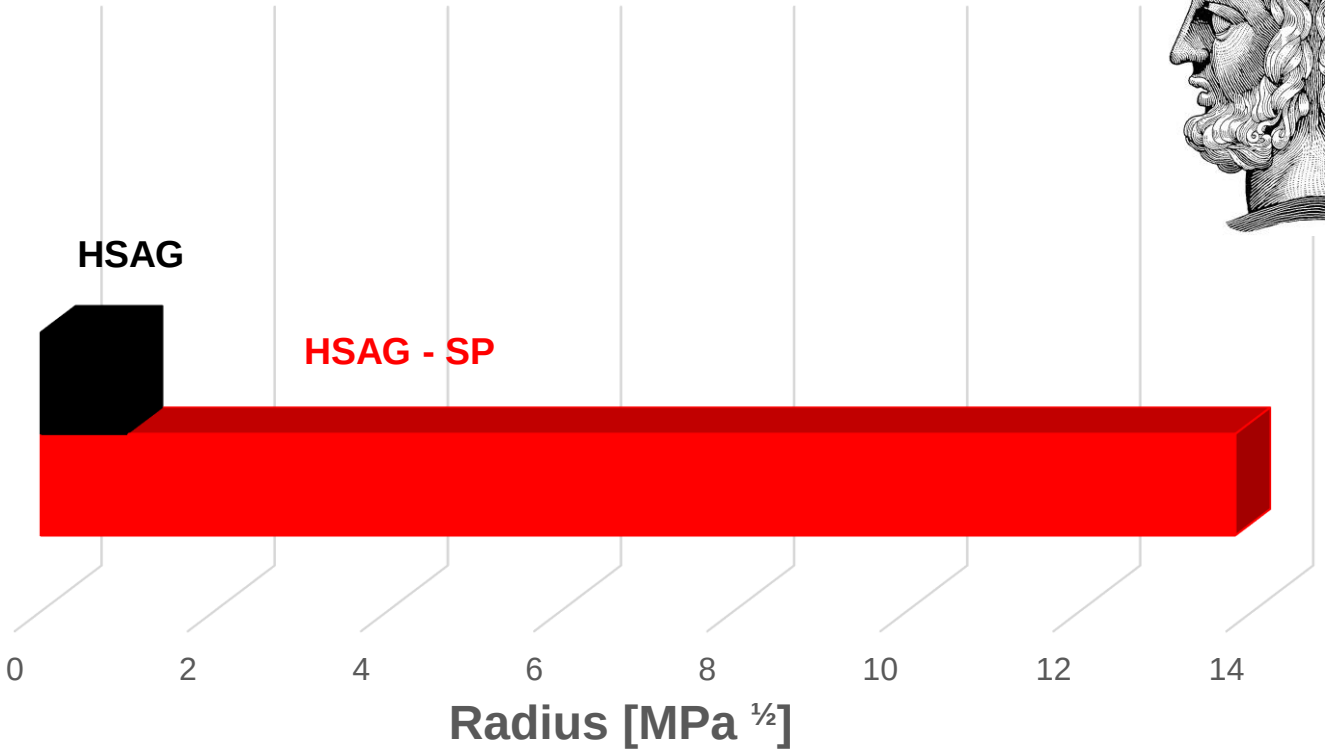
HSAG and HSAG/SP - Hansen sphere radius comparison



(*) Amount of SP on CA: 10 mass%

C6 building blocks

Sugars from hydrolyzed biomass

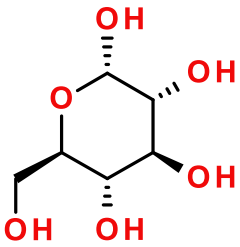
Biomass
for energy uses

Bio-ethanol

Biomass
for materials

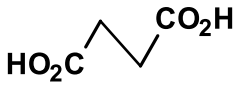
Polysaccharides

Chemicals from sugar

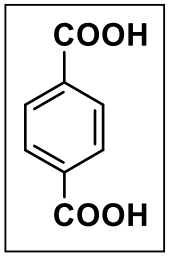


D-glucose

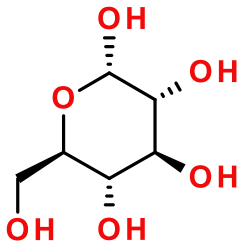
Chemicals from sugar - Target molecules



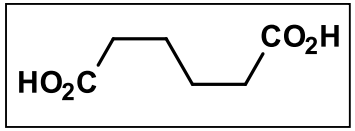
succinic acid



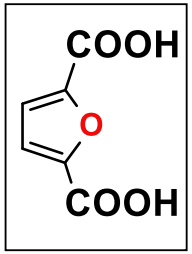
terephthalic acid



D-glucose

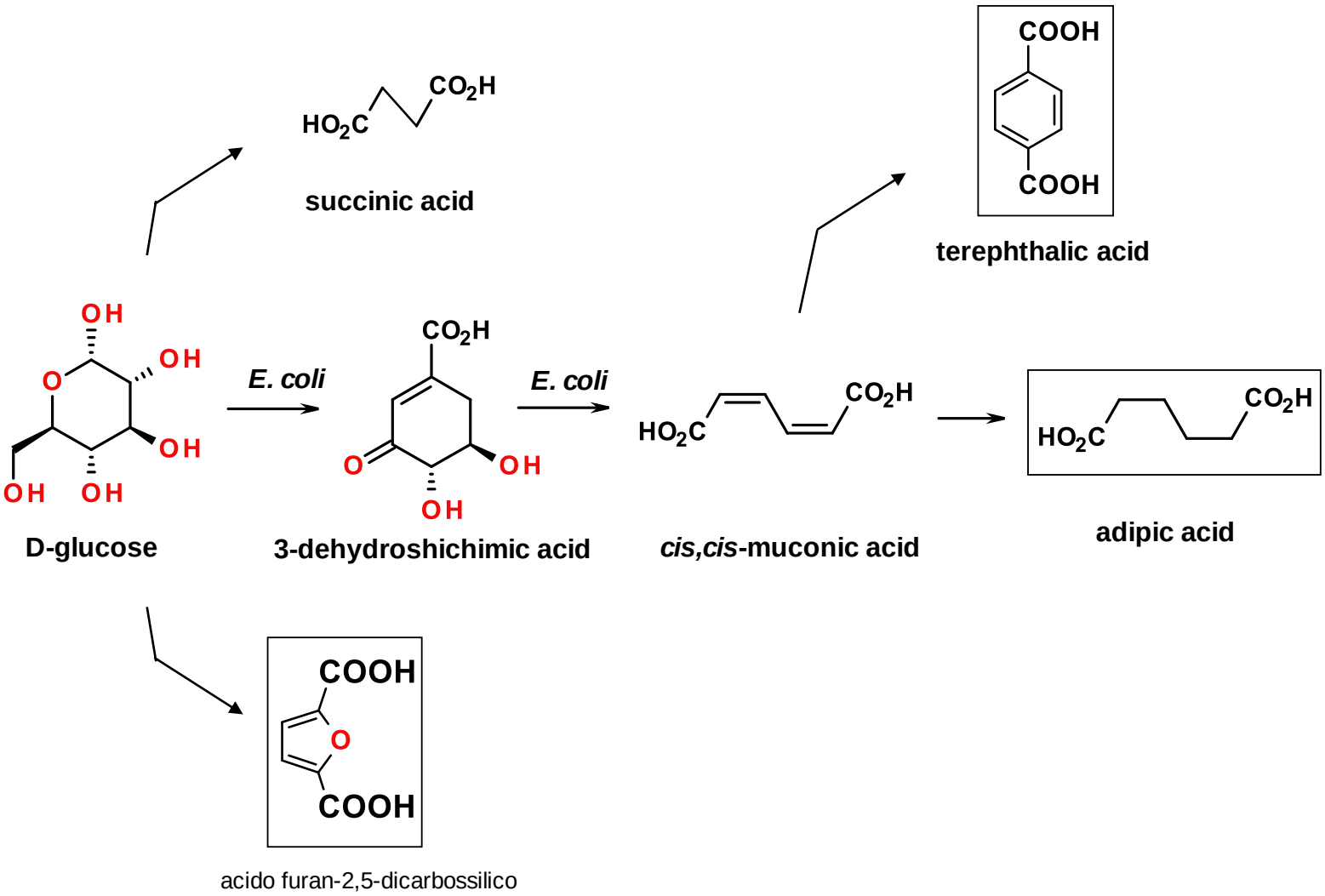


adipic acid

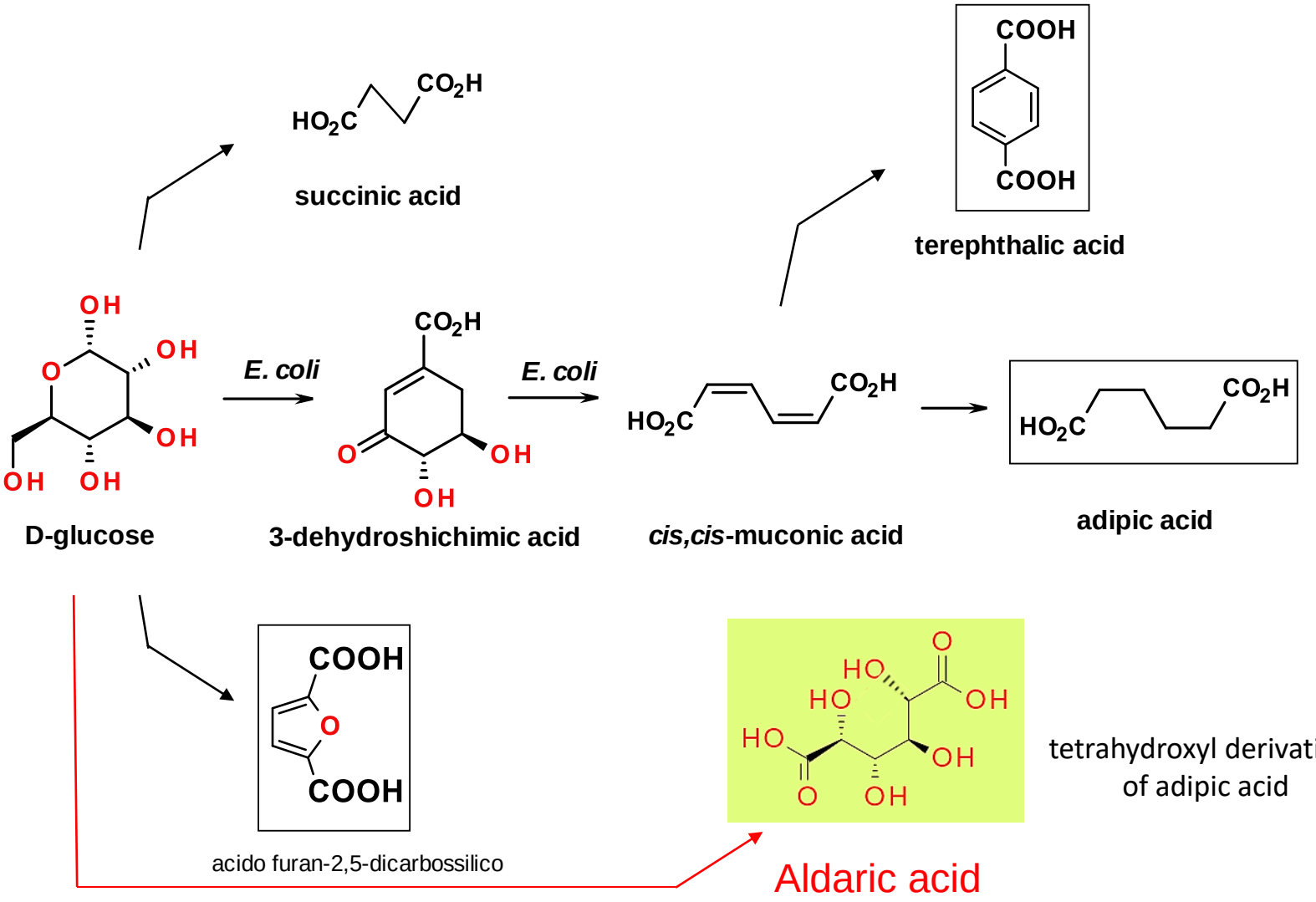


acido furan-2,5-dicarbossilico

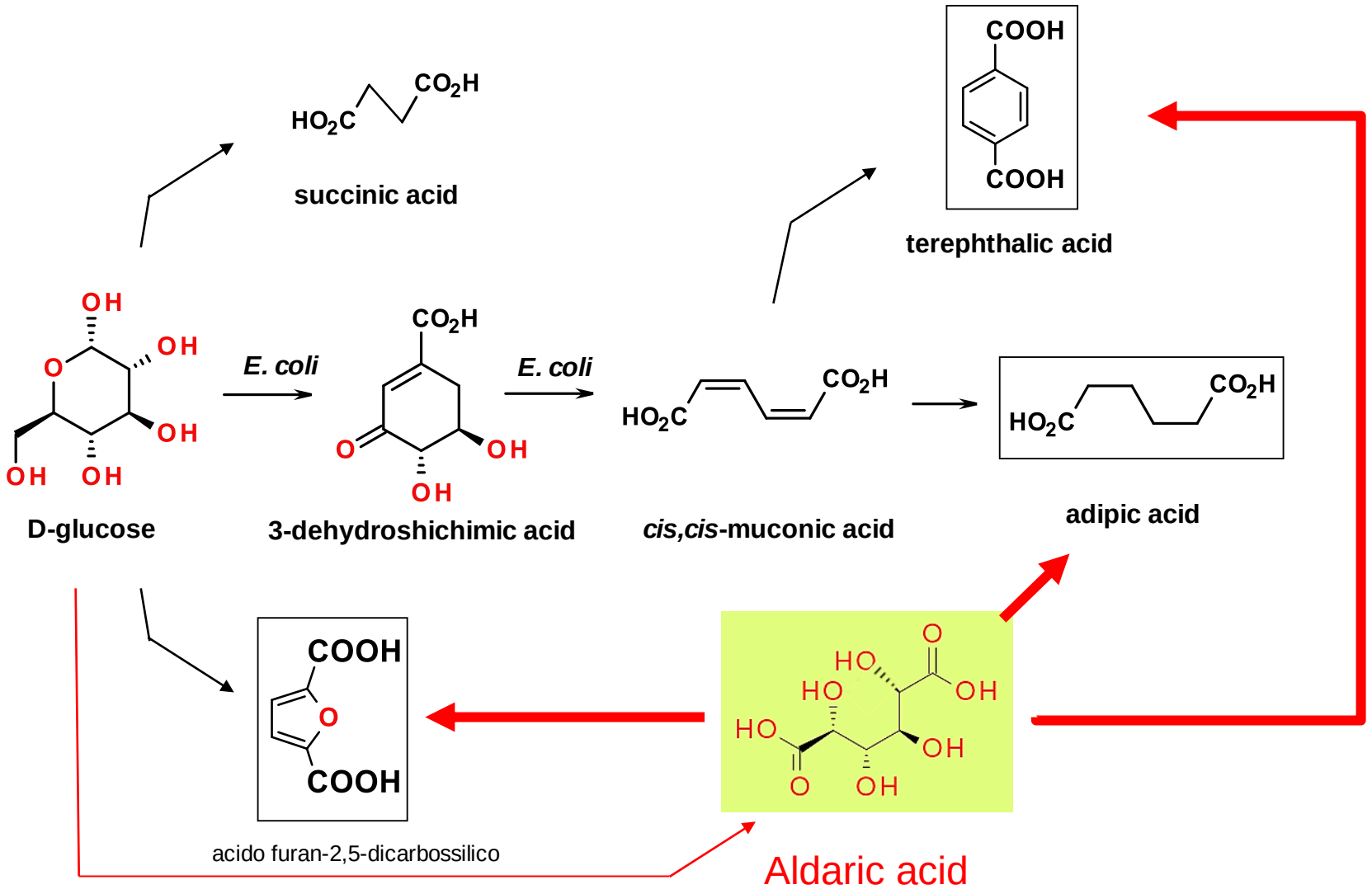
Chemicals from sugar - Target molecules



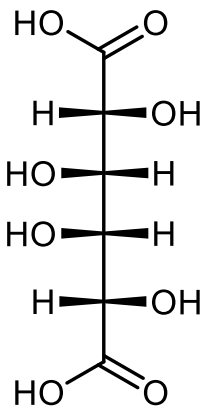
Dicarboxylic Acids from Hydrolyzed Biomasses



Dicarboxylic Acids from Hydrolyzed Biomasses

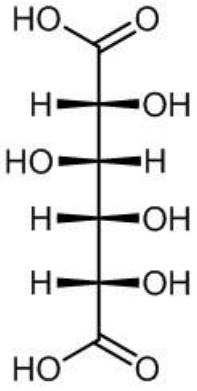


Aldaric acids as platform chemicals



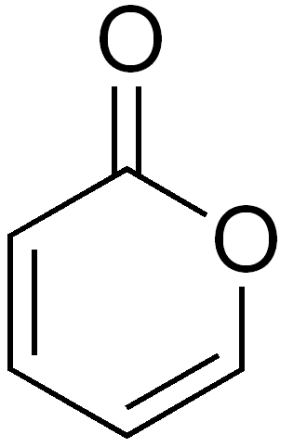
Mucic
(galactaric)

$C_6H_{10}O_8$

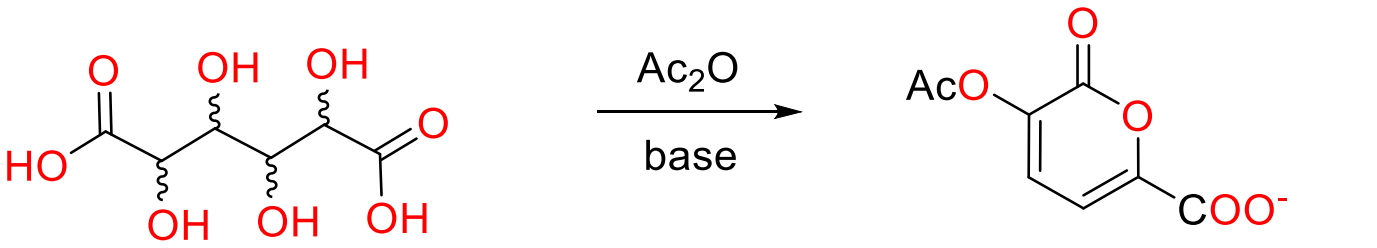


D-Glucaric

From aldaric acids to pyrones



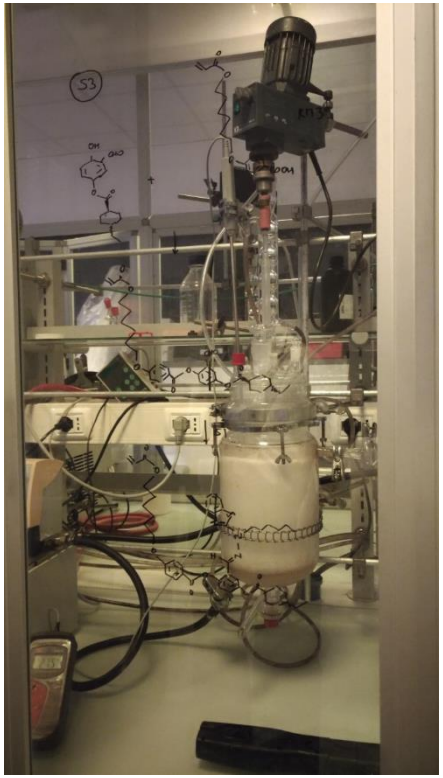
Synthesis of Pyrone Derivatives from Aldaric Acids @ ISCaMaP

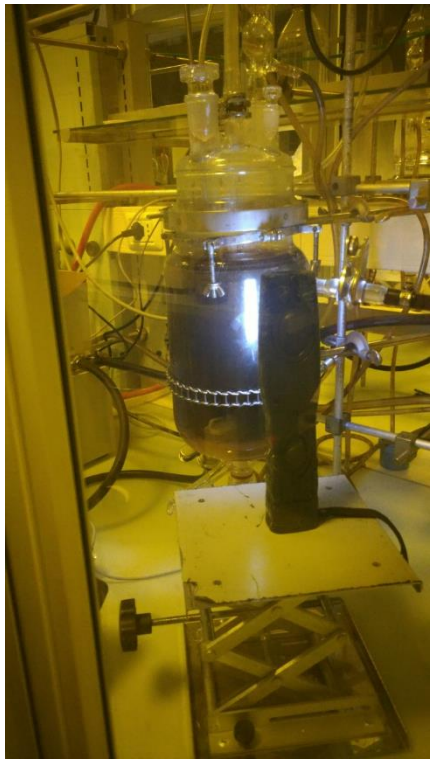


3-Acetoxy-2-oxo-2*H*-pyran-6-carboxylic salt (GAB-OAc)

- Easy procedure
- No solvent
- No catalyst
- High Conversion
- High Atom efficiency

Synthesis of Pyrone - Scale up





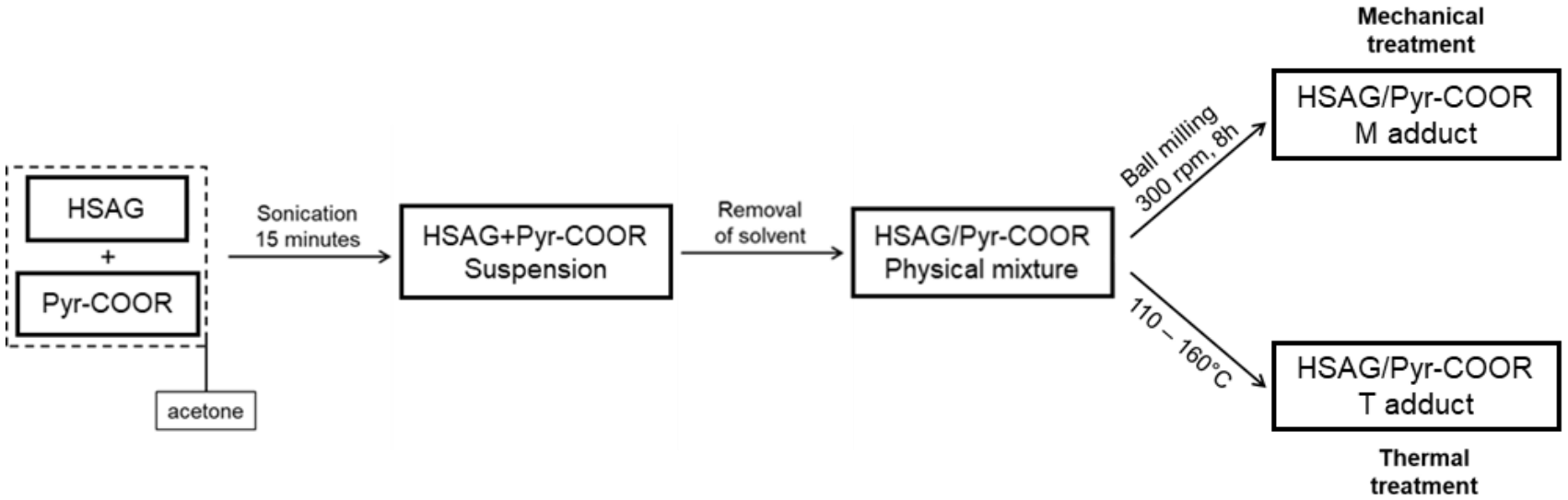
One Pot

2 hours

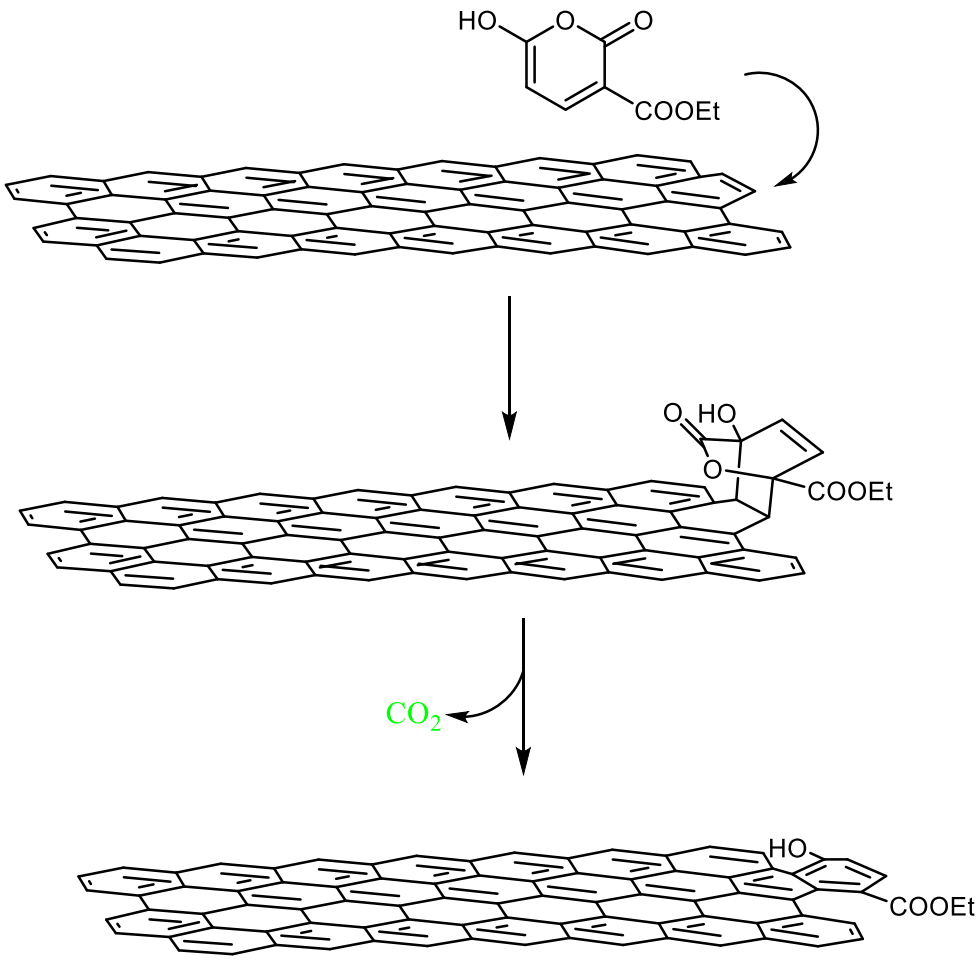
Yield = 75%

Functionalization of sp^2 carbon allotropes

Functionalization of HSAG with a Pyrone derivative



EDGE-GO - Functionalization of HSAG with a Pyrone derivative



Catalysis @POLIMI

Catalysis @POLIMI

- Graphene-based waterborne nanoreactors for the confinement of organic synthesis
- Ruthenium decoration of sp^2 carbon allotropes
- Silver decoration of sp^2 carbon allotropes

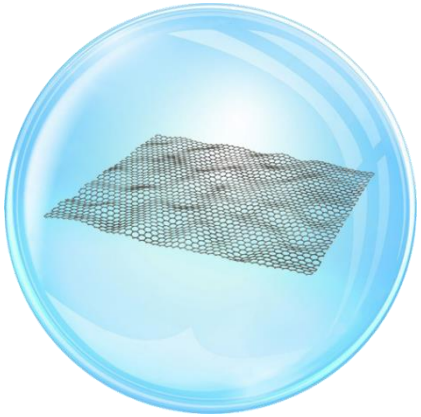
Graphene-based waterborne nanoreactors

for the confinement of organic synthesis

Waterborne nanoreactors based on graphene layers

Why graphene layers?

- ☞ High surface area
- ☞ π – electron density

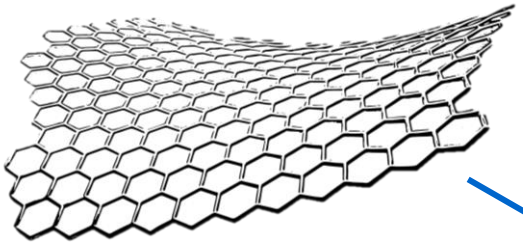


Why nanoreactors in water?

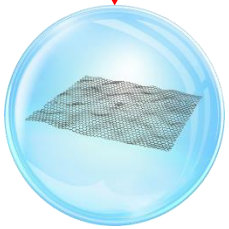
- ☞ To make reactions in confined space
- ☞ To improve the yields
- ☞ To avoid the use of toxic solvents
- ☞ To carry out more sustainable reactions

The Nanoreactor

Covalent functionalization with SP

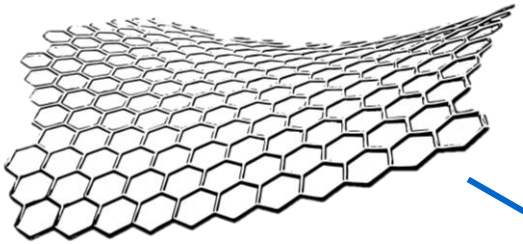


Supramolecular interaction with pyrrole containing polymeric surfactants

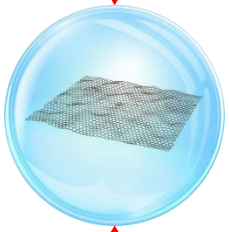


The Nanoreactor

Covalent functionalization with SP



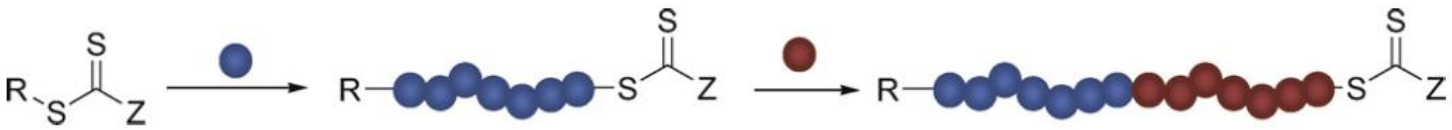
Supramolecular interaction with pyrrole containing polymeric surfactants



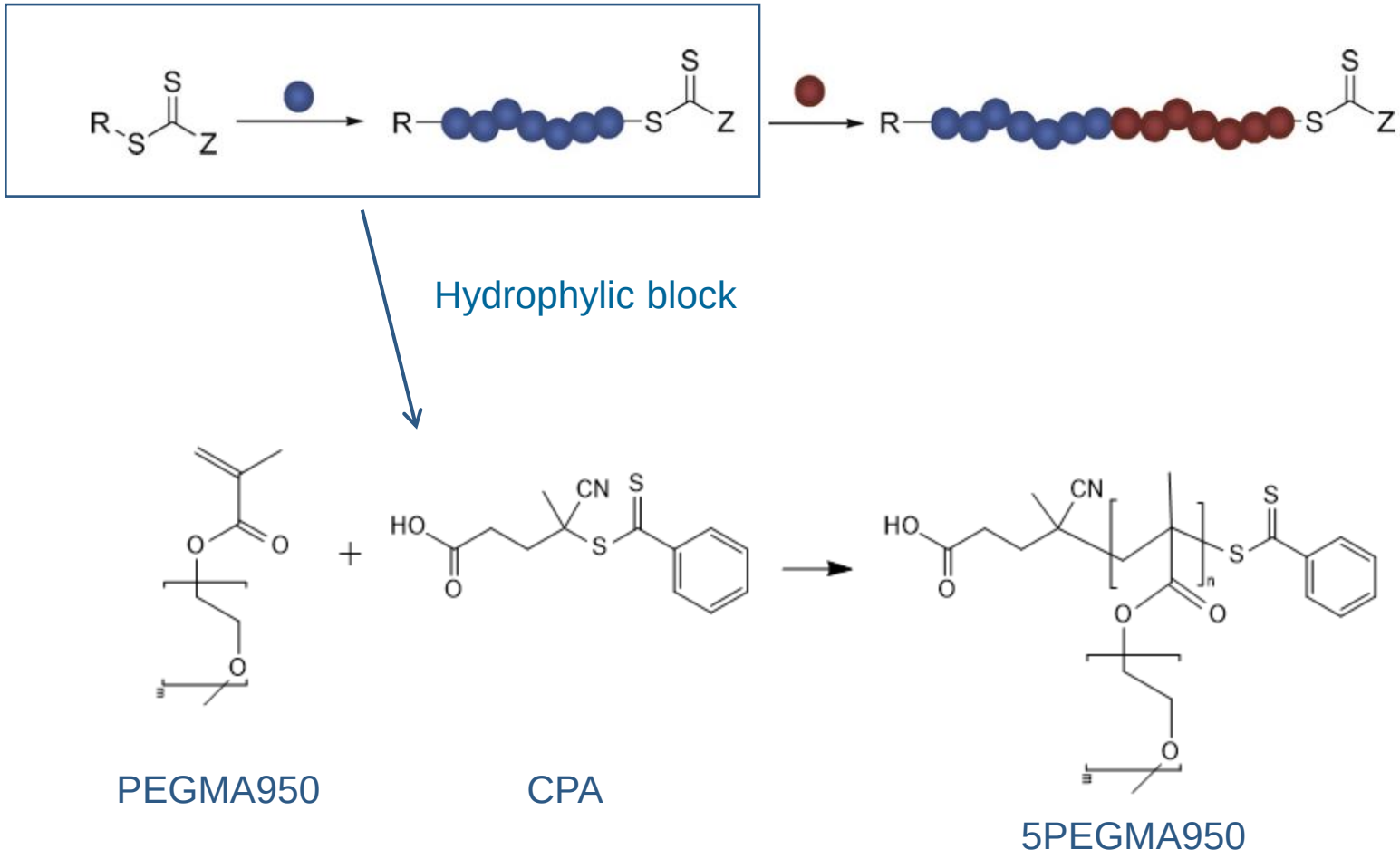
Lipophilic microenvironment and catalytic component

Confined space

Amphiphilic block copolymers via RAFT polymerization



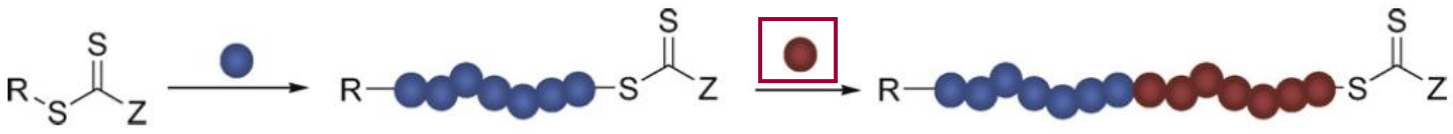
Amphiphilic block copolymers via RAFT polymerization



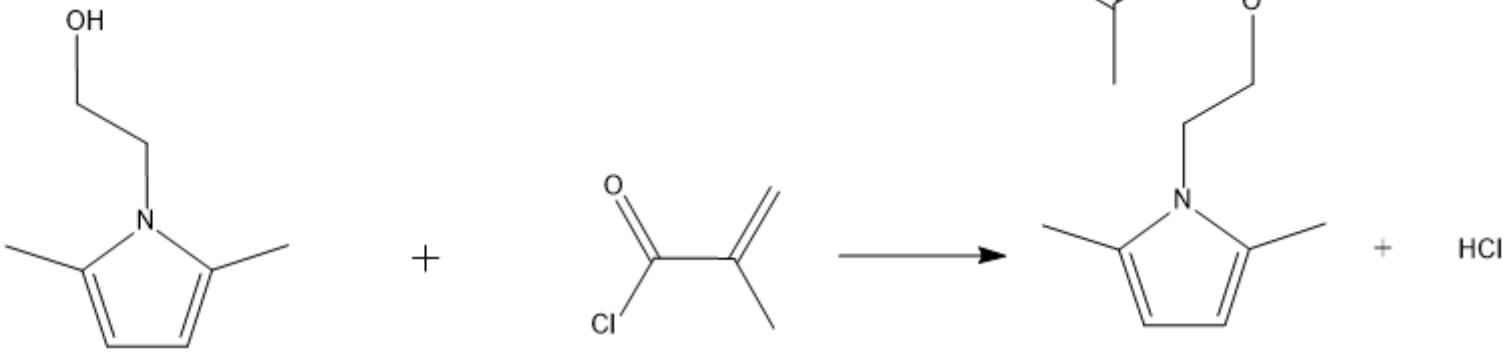
PEGMA950 = Poly(ethylene glycol)methyl ether methacrylate

CPA = 4-cyano-4 (phenylcarbonothioylthio)-pentanoic acid

Amphiphilic block copolymers via RAFT polymerization



The monomer for the lipophilic block



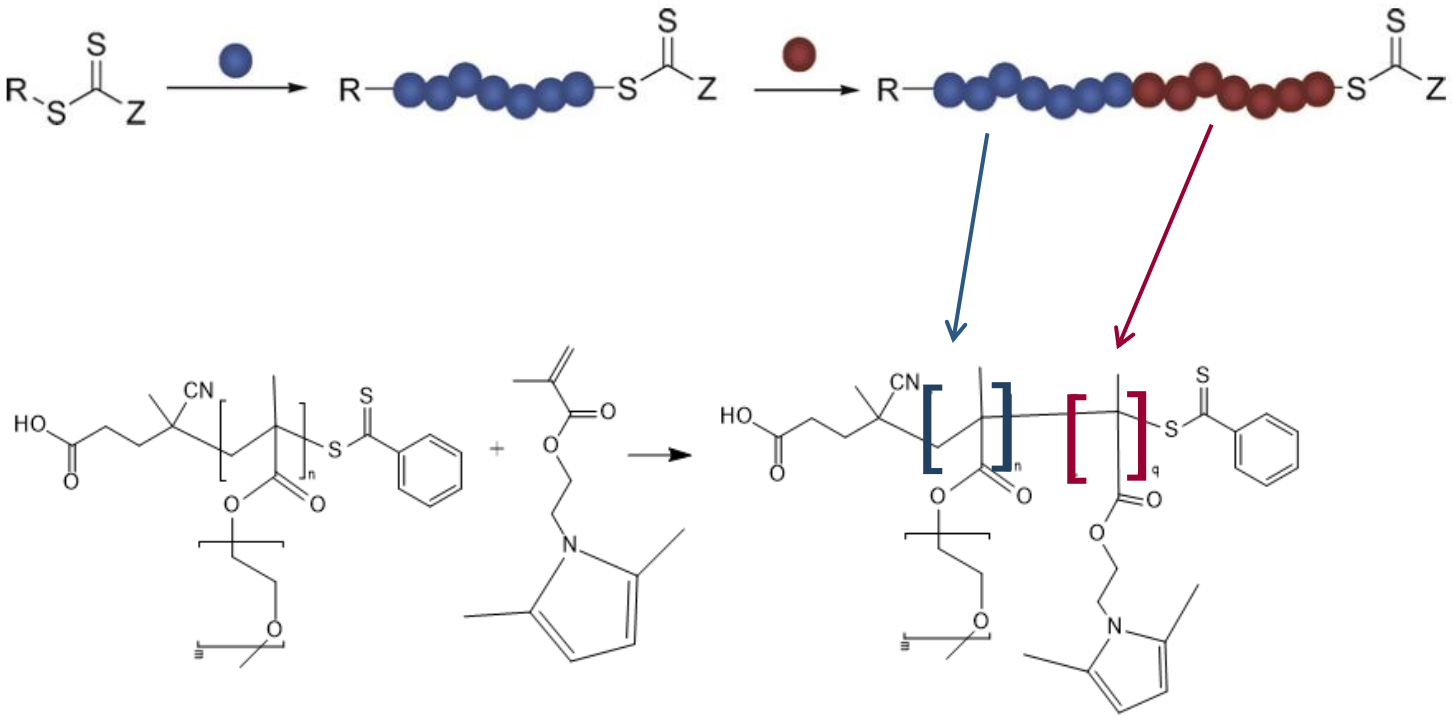
ethanol pyrrole (EP)

methacryloyl chloride

EPMA

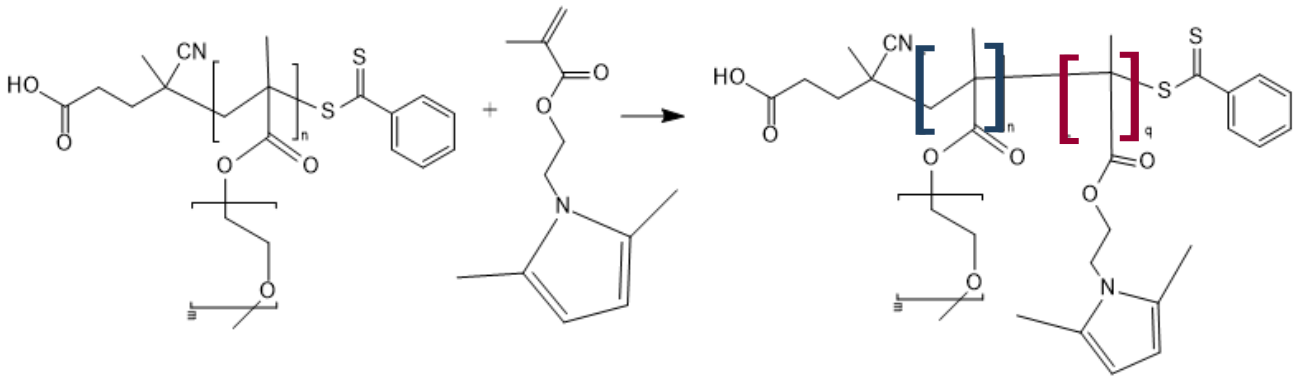
EPMA = 2-(2,5-dimethyl-1*H*-pyrrol-1-yl) ethyl methacrylate

Amphiphilic block copolymers via RAFT polymerization

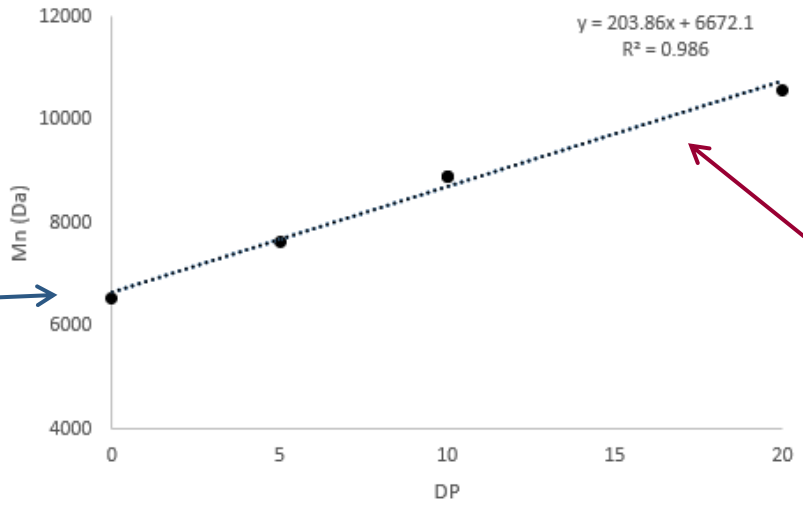


5PEGMA950-p(EPMA)

Amphiphilic block copolymers via RAFT polymerization



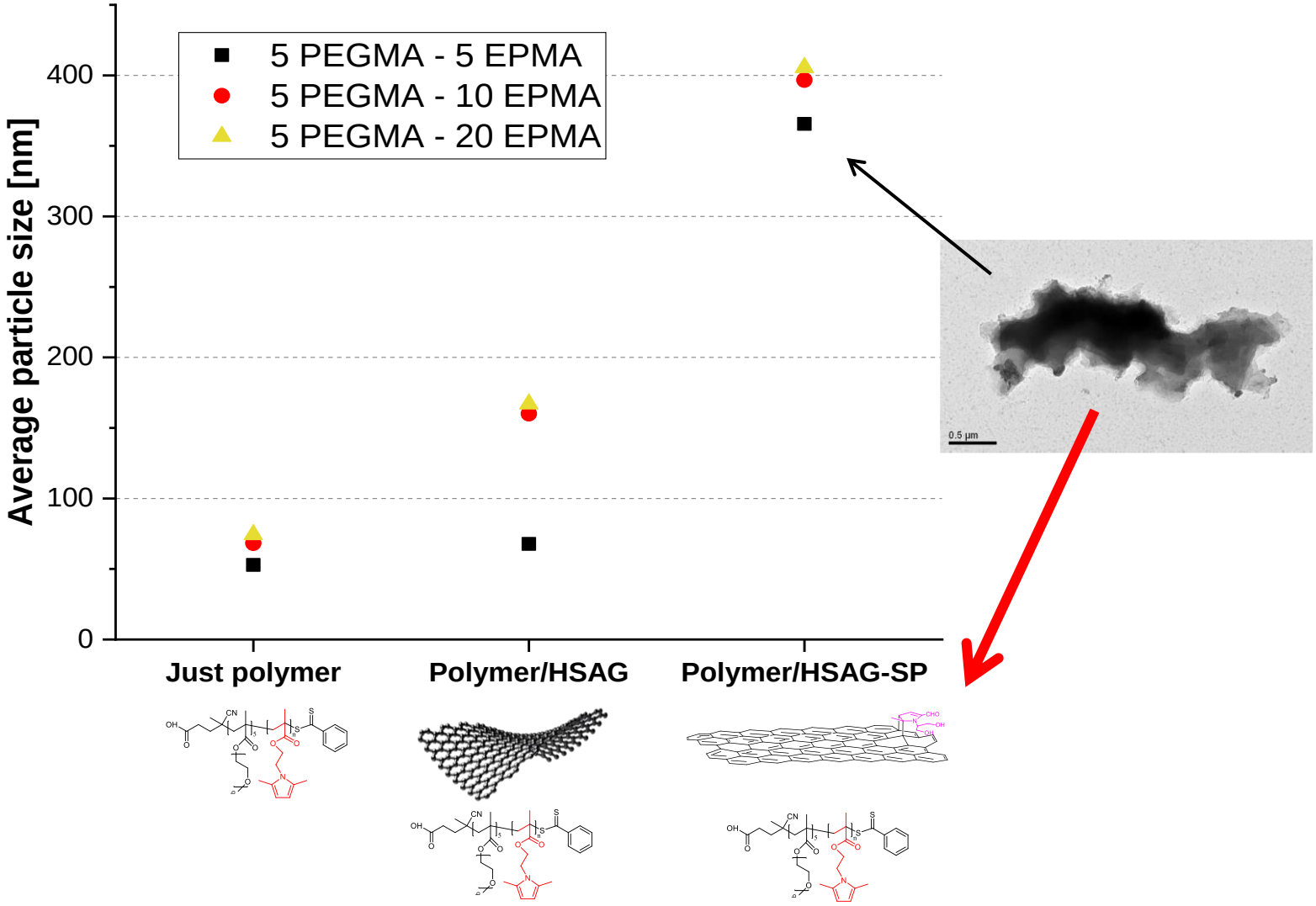
5PEGMA950-p(EPMA)



5PEGMA950

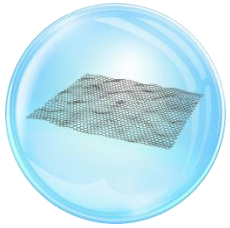
p(EPMA)

Formation of micelle-like sistems



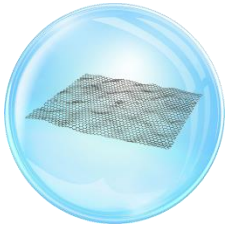
The adduct between 5PEGMA950-p(EPMA) and graphene layers as nanoreactor for organic synthesis

The *Dropcat catalyst*



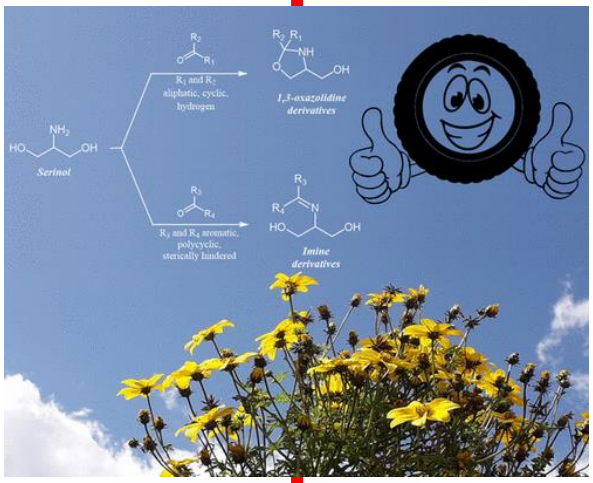
Synthesis of imines with the *Dropcat catalyst*

Why imines?



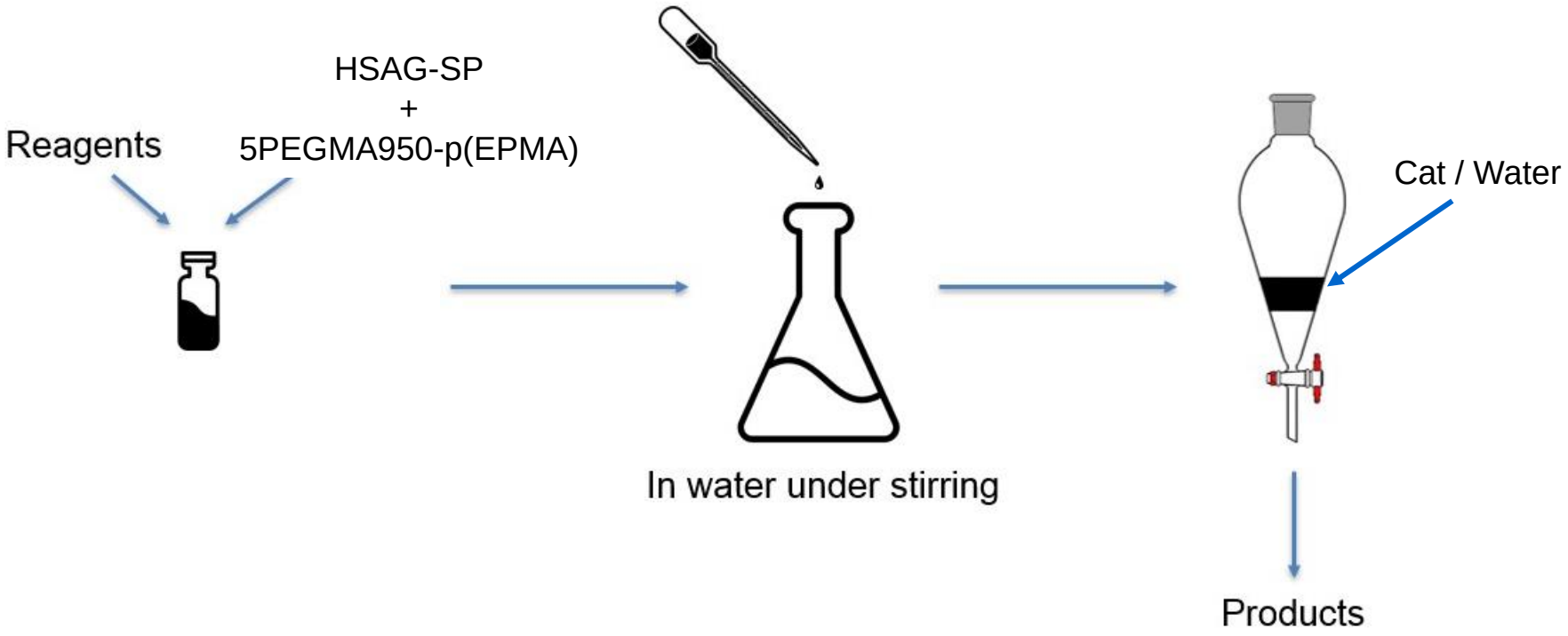
☞ They can be used in vulcanization systems of tyre compounds

Large scale applications

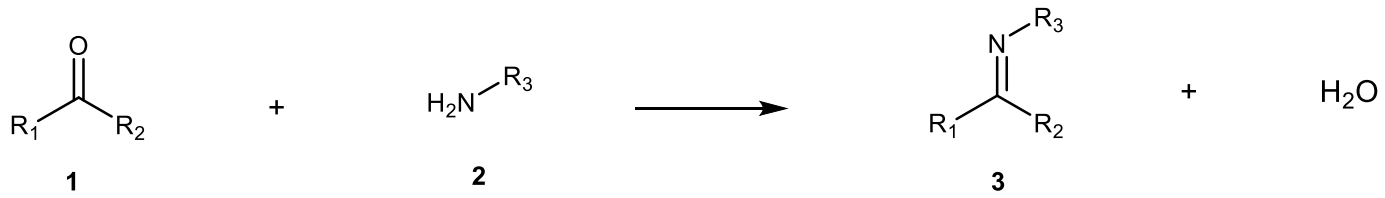


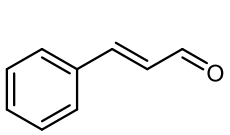
as secondary accelerators
in silica based compounds

Synthesis of imines with the *Dropcat catalyst*

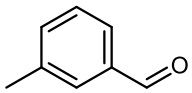


Synthesis of imines with the *Dropcat catalyst*

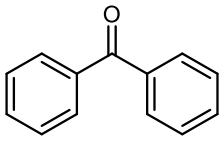




1a



1b



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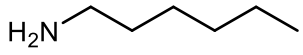

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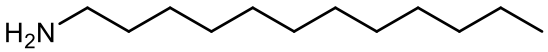

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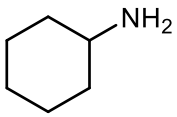
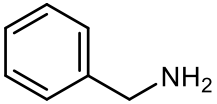
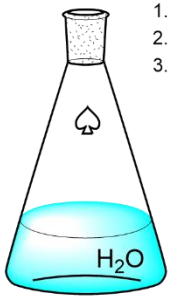


2c



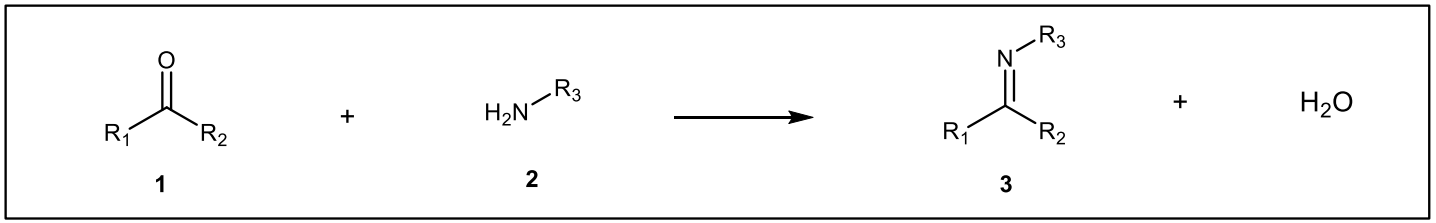
2d

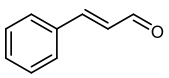
Just 5 minutes!



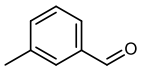
1. HSAG-SP/PEG-EP
2. Amine
3. carbonyl compound

Synthesis of imines with the *Dropcat catalyst*

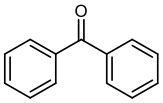




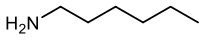
1a



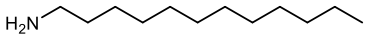
1b



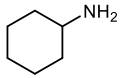
1c



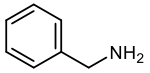
2a



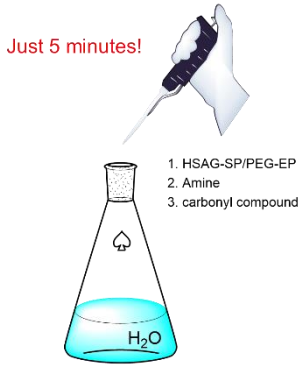
2b



2c

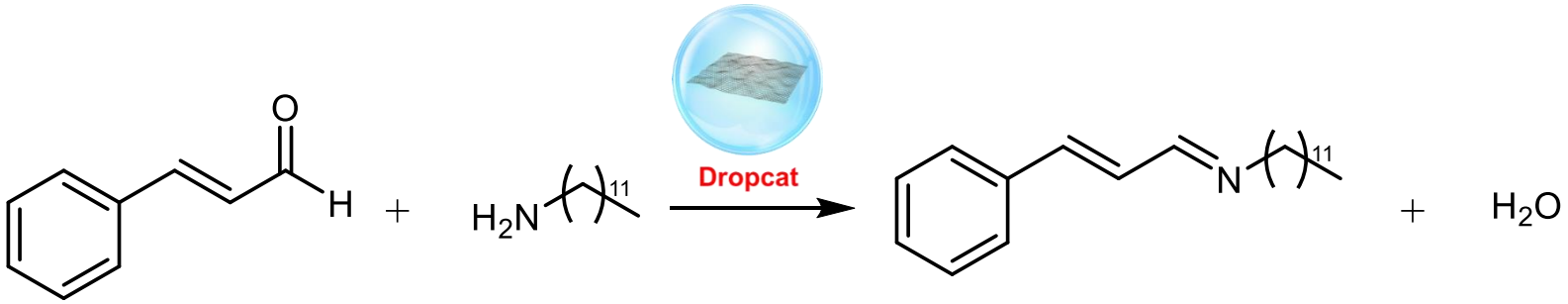


2d

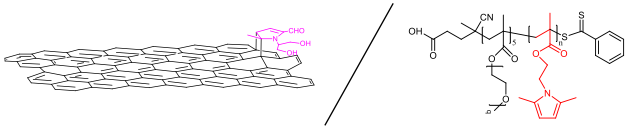


entry	reag.[a]	time (min)	T (°C)	Cat.	yield (%)
1	1a + 2a	240	180	-	60
3	1a + 2a	120	r.t.	Drop-cat	91
12	1a + 2b	120	r.t.	Drop-cat	80
13	1a + 2c	120	r.t.	Drop-cat	81
14	1a + 2d	120	r.t.	Drop-cat	72
15	1b + 2a	120	r.t.	Drop-cat	82
16	1c + 2a	120	r.t.	Drop-cat	10

Synthesis of imines with the *Dropcat catalyst*



time (min)	Selected system	T (°C)	Yield (%)
240	-	180	60
5	5PEGMA-5EPMA/HSAG-SP	25	98



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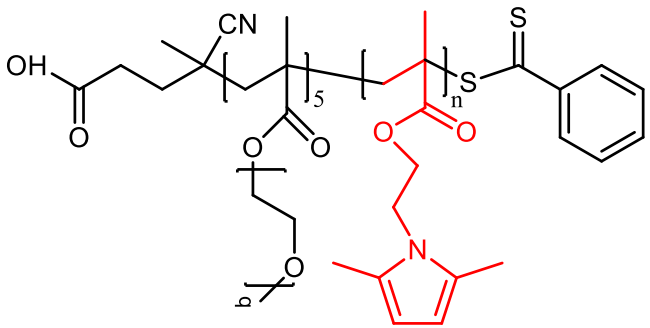
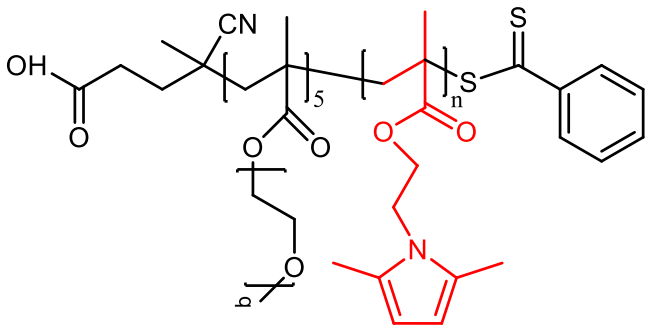
What is the role of the monomer?

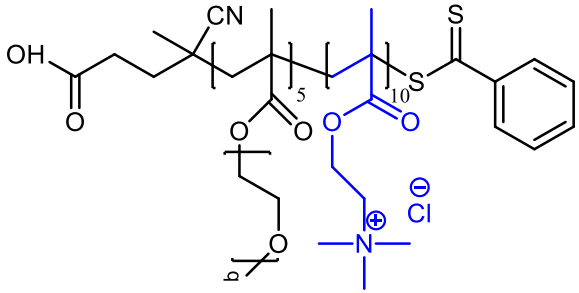


5PEGMA-5EPMA

What is the role of the monomer?

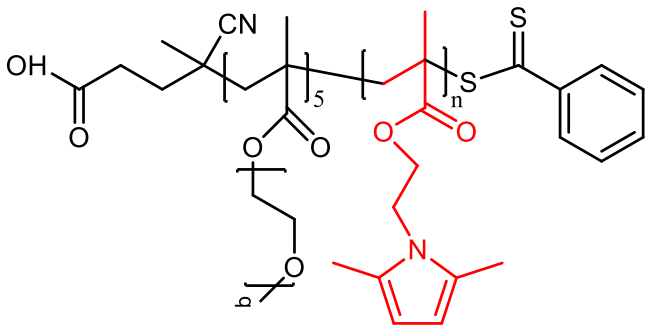


5PEGMA-5EPMA

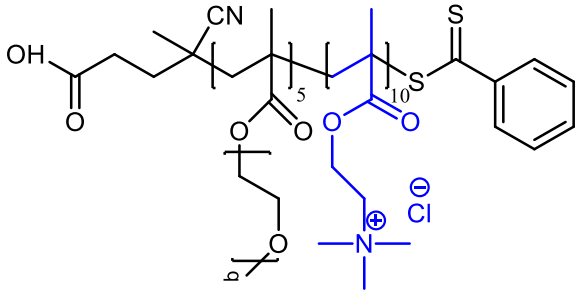


5PEGMA-10TMAEMA

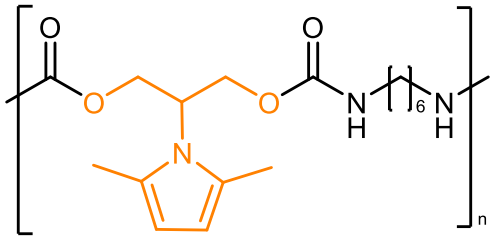
What is the role of the monomer?



5PEGMA-5EPMA



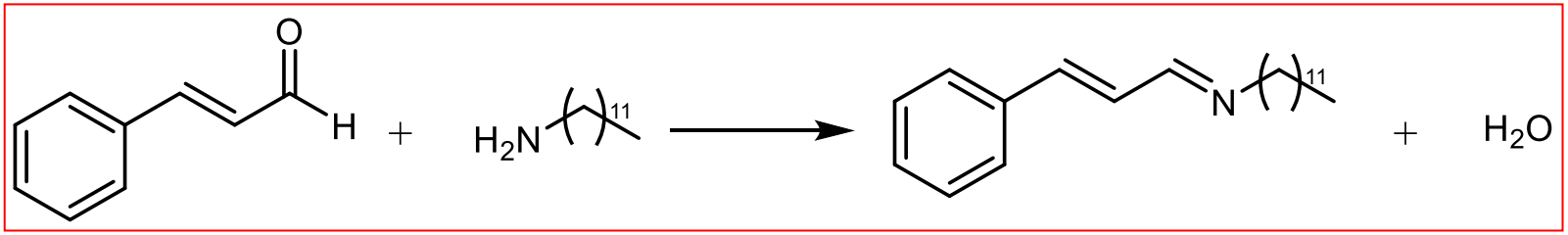
5PEGMA-10TMAEMA

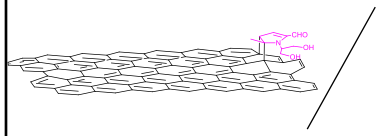
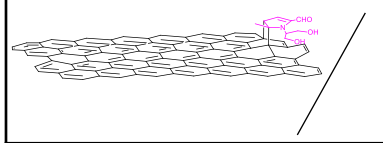
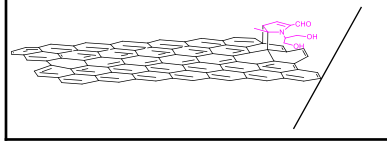


PU-SP

Mn = 1430 Da

The role of the monomer. Dropcat catalysts with different polymers



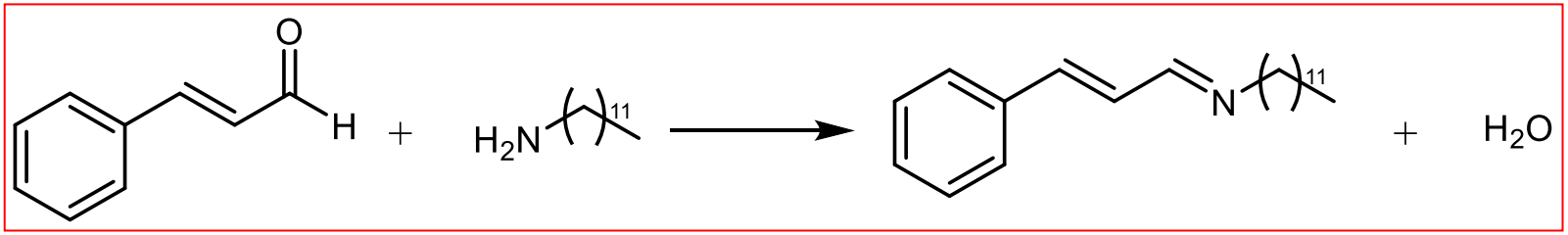
Selected system	Yield (%)
 5PEGMA-5EPMA/HSAG-SP	**98**
 5PEGMA-TMAEMA/HSAG-SP	23
 PU-SP/HSAG-SP	52

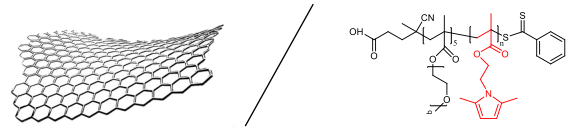
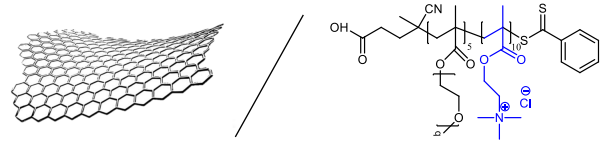
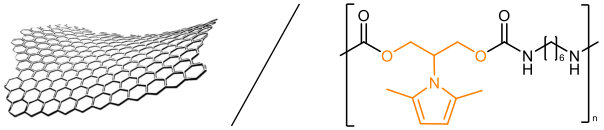
Temperature = 25°C

time = 5 minutes

U. Capasso Palmiero, M. Sponchioni, F. Margani, D. Moscatelli, M. Galimberti, V. Barbera *Small, 16(40), 2001207 (2020)*

The role of graphene layers. Dropcat catalysts with pristine nanographite



Selected system	Yield (%)
 5PEGMA-5EPMA/HSAG	-
 5PEGMA-TMAEMA/HSAG	30
 PU-SP/HSAG	50

Temperature = 25°C

time = 5 minutes

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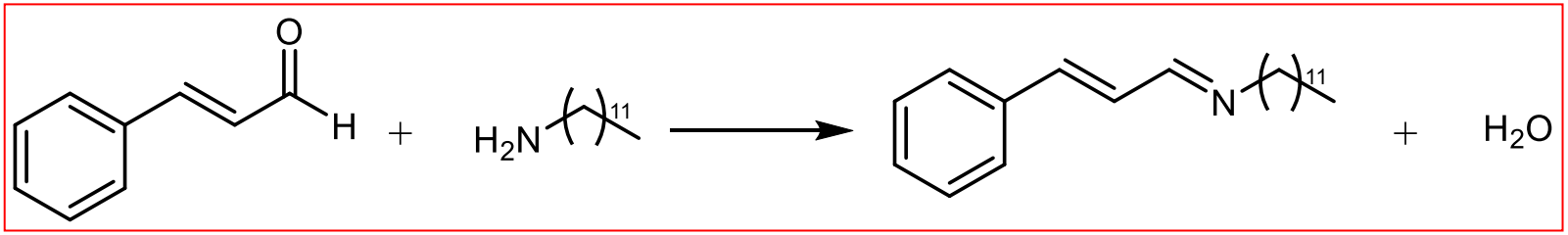
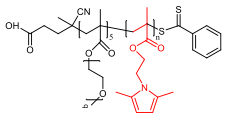
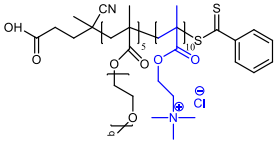
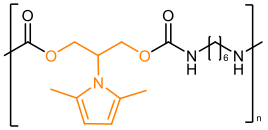
The role of graphene layers. Dropcat catalysts without graphene layers



Selected system	Yield (%)
 5PEGMA-5EPMA	10
 5PEGMA-TMAEMA	23
 PU-SP	53

Temperature = 25°C

time = 5 minutes

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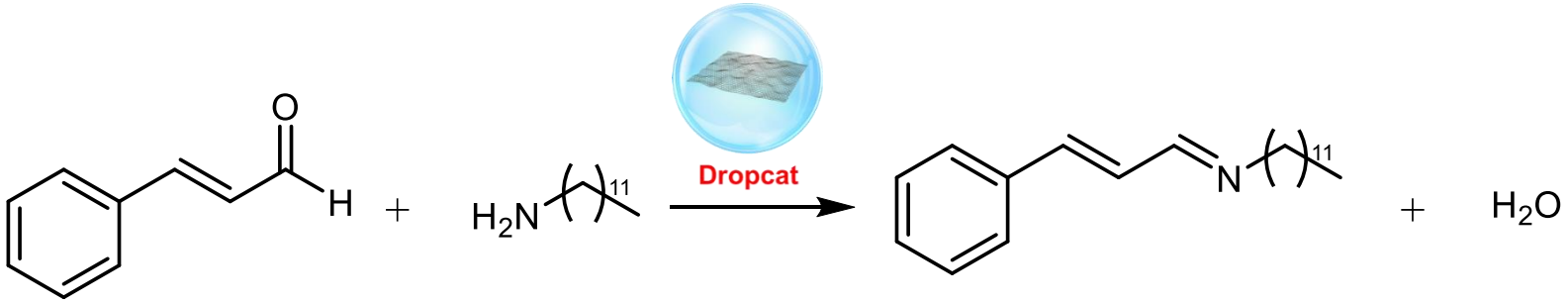
The synergy of covalent and supramolecular functionalization



time (min)	Selected system	T (°C)	Yield (%)
240	-	180	60
5	5PEGMA-5EPMA	25	10
5	5PEGMA-5EPMA/HSAG	25	-
5	**5PEGMA-5EPMA/HSAG-SP**	**25**	**98**

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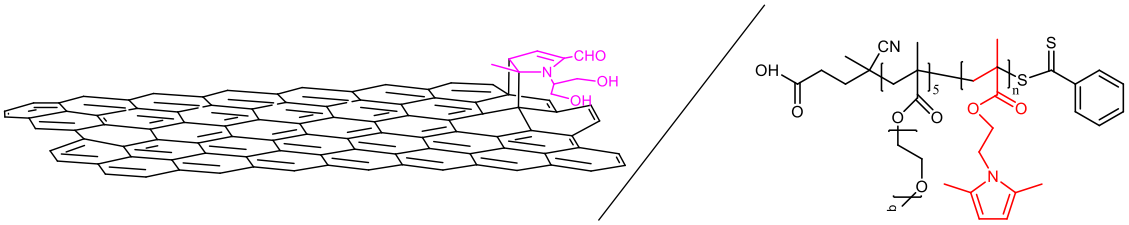
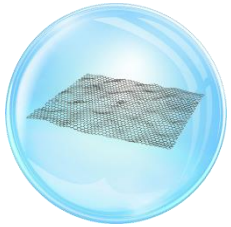
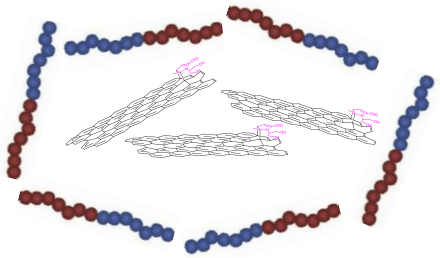
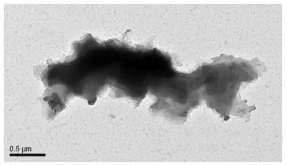
Recyclability and catalytic activity

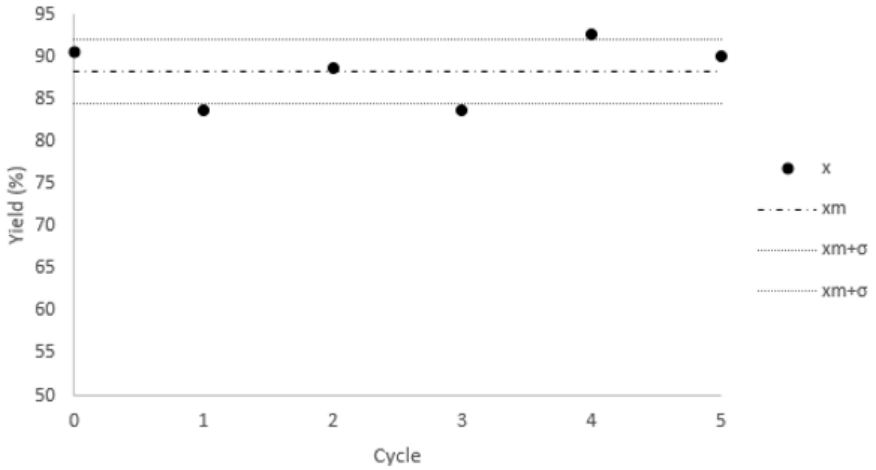
5PEGMA-5EPMA/HSAG-SP







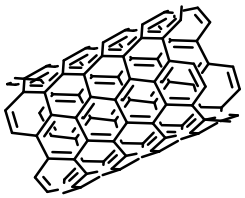


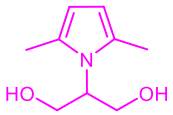


EPMA based polymer reusability

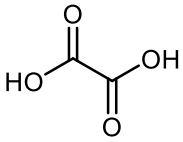
Ruthenium decoration of sp^2 carbon allotropes

Ruthenium decoration of sp^2 carbon allotropes

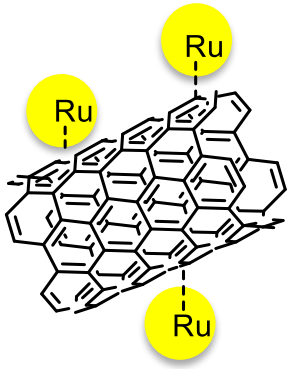


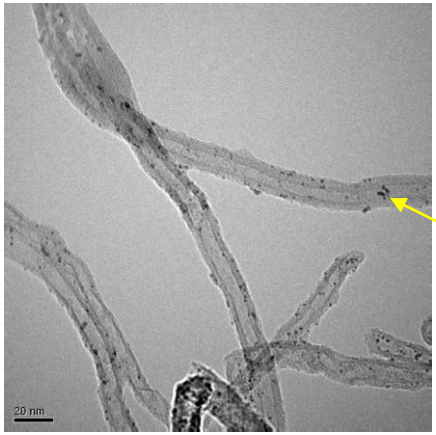


$RuCl_3$



Sustainable reduction





Nanoparticles

≥ 12 Å

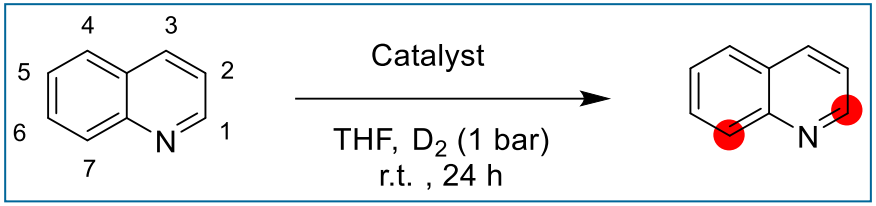
Italian Patent Application n. 102020000020113 filed on 13 August 2020, Inventors: V. Barbera, M. Galimberti, G. Candiani.
Italian Patent Application n. 102020000020104 filed on 13 August 2020, Inventors: V. Barbera, M. Galimberti, G. Pièters; A. Palazzolo

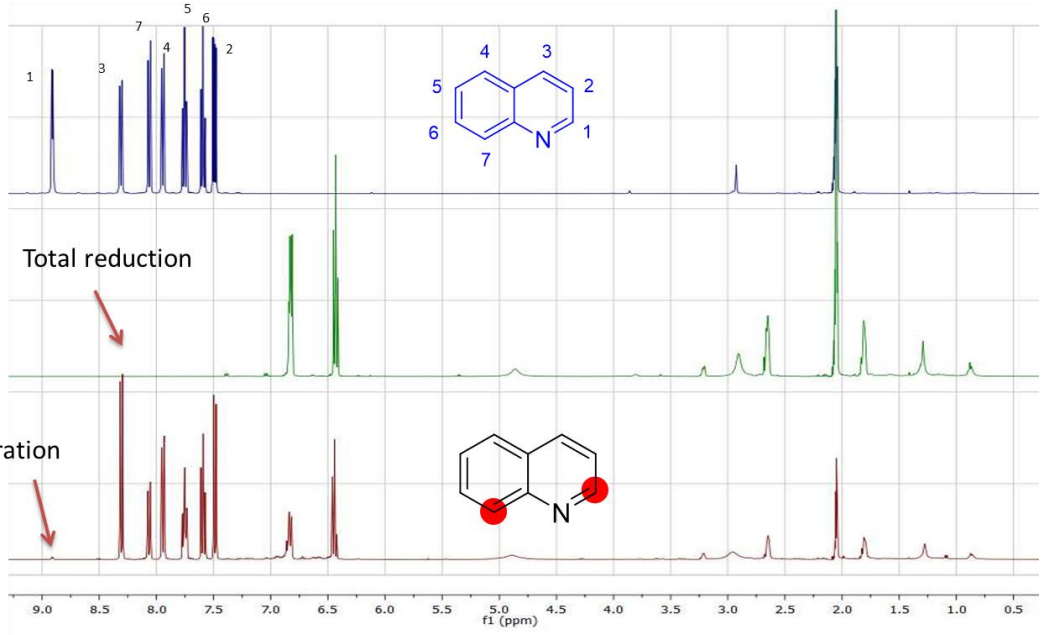
Ruthenium decoration of sp^2 carbon allotropes

Deuteration of organic molecules

Hydrogen/Deuterium exchange: high selectivity and conversion,

If compared with a commercial one



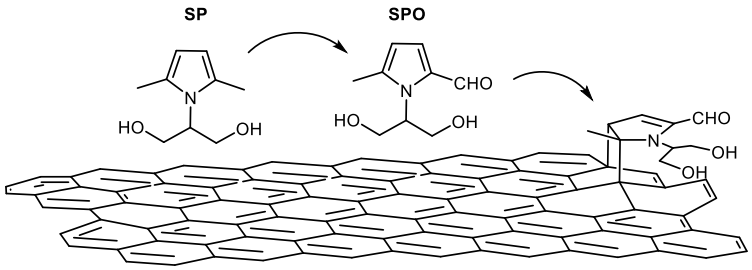


Italian Patent Application n. 102020000020113 filed on 13 August 2020, Inventors: V. Barbera, M. Galimberti, G. Candiani.
Italian Patent Application n. 102020000020104 filed on 13 August 2020, Inventors: V. Barbera, M. Galimberti, G. Pièters; A. Palazzolo

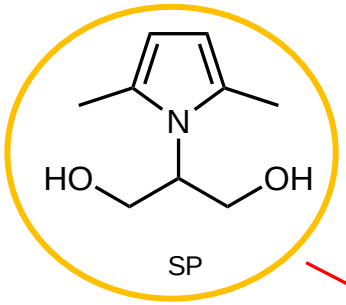
Silver decoration of sp^2 carbon allotropes

Silver decoration of sp^2 carbon allotropes

sp^2 carbon allotropes and silver decorated sp^2 carbon allotropes



Diels - Alder cycloaddition reaction mechanism



Dispersability enhancer

Allows the use of **dH_2O** as solvent for a multitude of applications (e.g. **coatings**, **paintings**, ecc…)

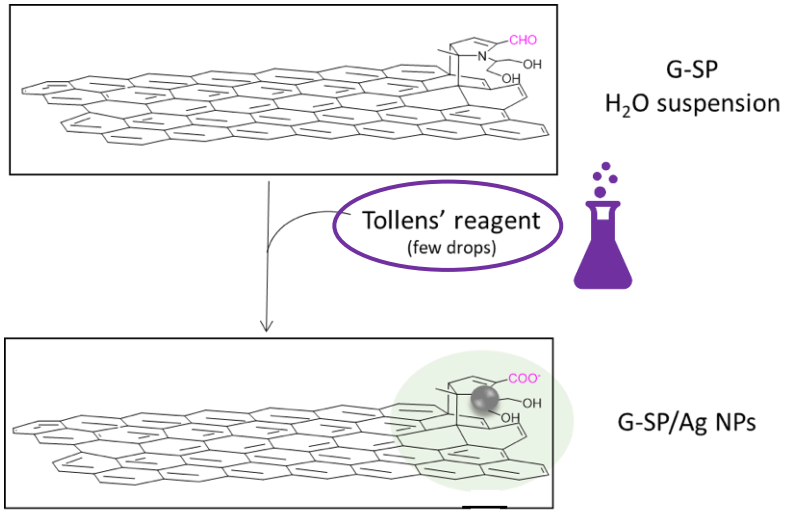
Ag^+ reduction to Ag

Ag NPs linking

[**V. Barbera, A. Bernardi, A. Palazzolo, A. Rosengart, L. Brambilla, M. Galimberti,** Facile and Sustainable Functionalization of Graphene Layers with Pyrrole Compounds, Pure Appl. Chem., vol. 90, no. 2, pp. 253–270, **2018**]

Silver decoration of sp^2 carbon allotropes

Synthesis of the adducts: decoration with Ag NPs

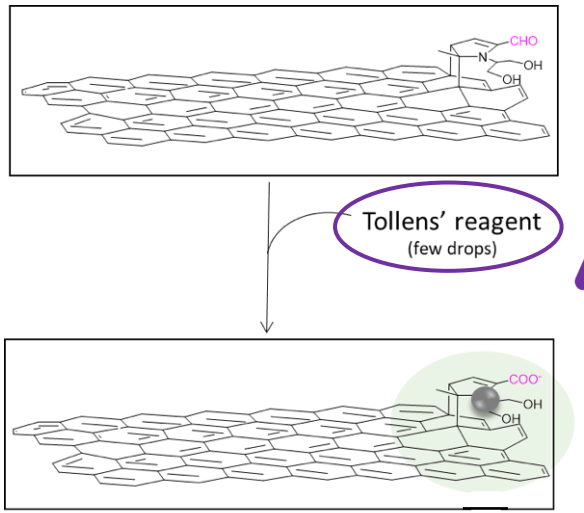


- ✓ Simple functionalization process
- ✓ Without reducing agents
- ✓ Sustainable process

Italian Patent Application n. 102020000020113 filed on 13 August 2020, Inventors: V. Barbera, M. Galimberti, G. Candiani.
Italian Patent Application n. 102020000020104 filed on 13 August 2020, Inventors: V. Barbera, M. Galimberti, G. Pièters; A. Palazzolo

Silver decoration of sp^2 carbon allotropes

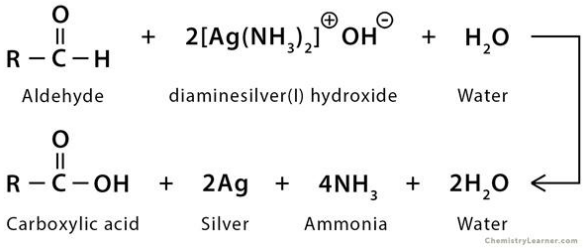
Synthesis of the adducts: decoration with Ag NPs



G-SP
H_2O suspension

G-SP/Ag NPs

Tollens' Test



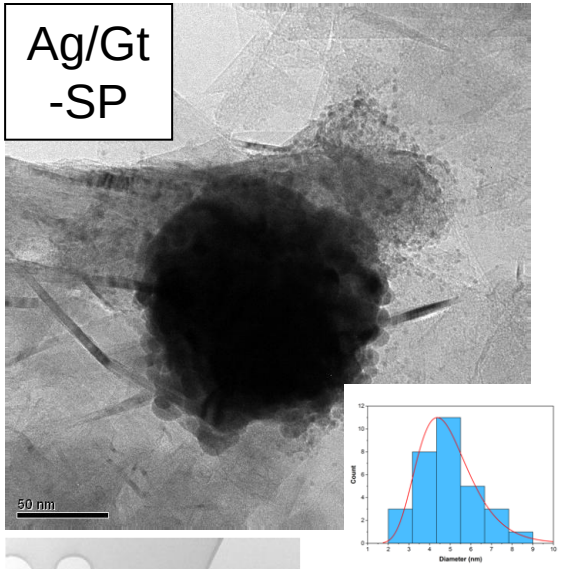
- ✓ Simple functionalization process
- ✓ Without reducing agents
- ✓ Sustainable process

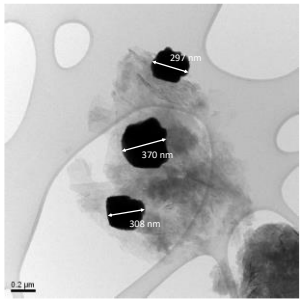
Italian Patent Application n. 102020000020113 filed on 13 August 2020, Inventors: V. Barbera, M. Galimberti, G. Candiani.
Italian Patent Application n. 102020000020104 filed on 13 August 2020, Inventors: V. Barbera, M. Galimberti, G. Pièters; A. Palazzolo

Silver decoration of sp^2 carbon allotropes

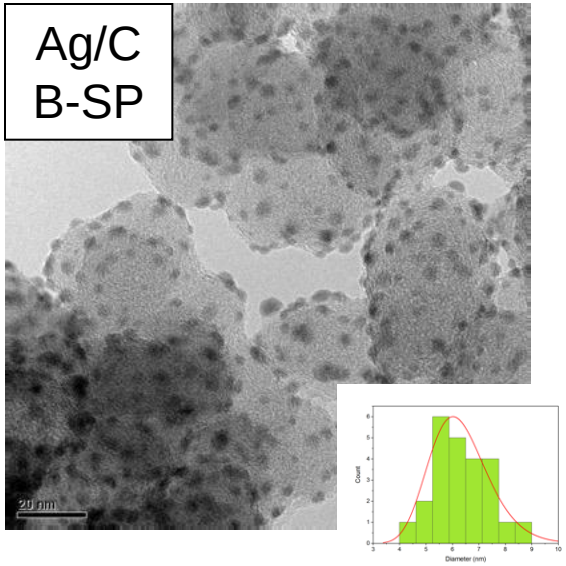
Synthesis of the adducts: decoration with Ag NPs

Ag/Gt -SP

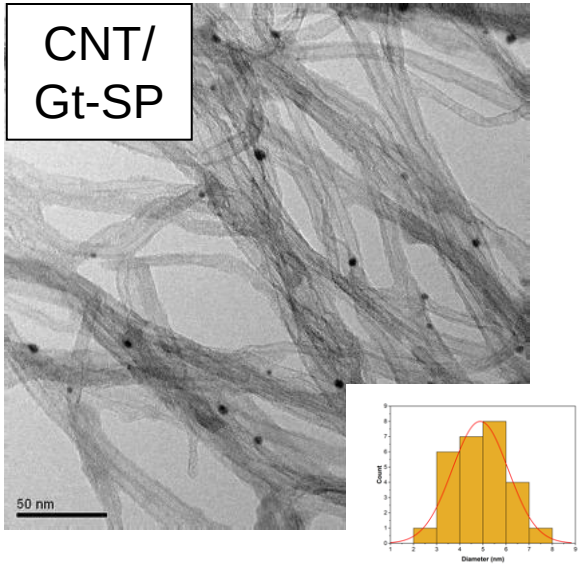




Ag/C B-SP



CNT/ Gt-SP



SAMM
SERVIZIO DI ANALISI MICROSTRUTTURALI DEI MATERIALI

Italian Patent Application n. 102020000020113 filed on 13 August 2020, Inventors: V. Barbera, M. Galimberti, G. Candiani.
Italian Patent Application n. 102020000020104 filed on 13 August 2020, Inventors: V. Barbera, M. Galimberti, G. Pièters; A. Palazzolo

Substrates from proteins: BSF

Organic urban waste

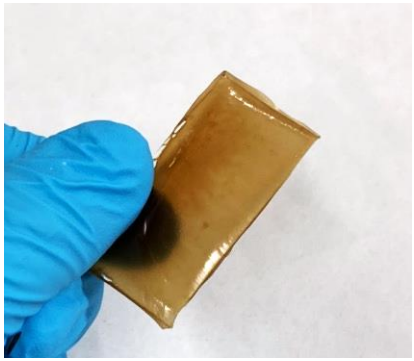
2020. Progetto RICH, Cariplo *grant* (2020) in collaboration with Università di Milano, Università dell'Insubria e DIK (Istituto tedesco della gomma)

Biobased substrates and monomers for printed electronics

Organic urban waste

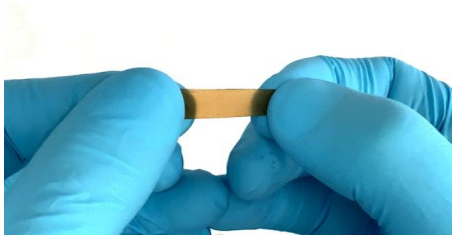
Hermetia illucens (black soldiers fly **larvae**)

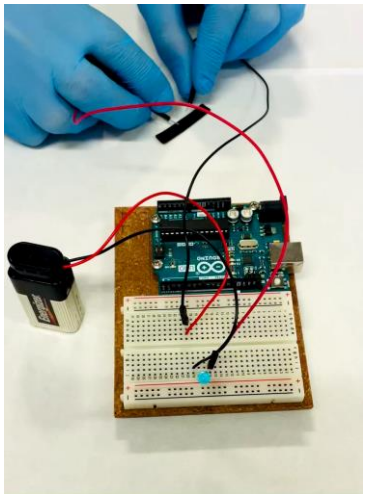
BSF proteins



high flexibility and compostability

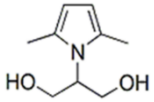
BSF protein extract will be characterized by means of proteomic techniques
BCA; SDS-PAGE; LC-MS (Mass Spectrometry)



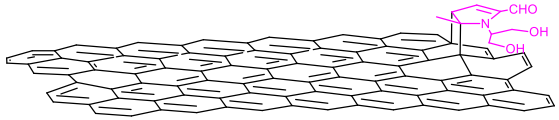


Conclusions

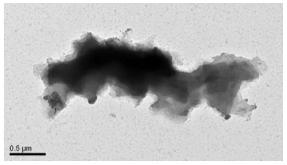
- Biosourced *Janus* molecule



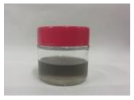
- The functionalization of sp^2 carbon allotropes



- Waterborne nanoreactors

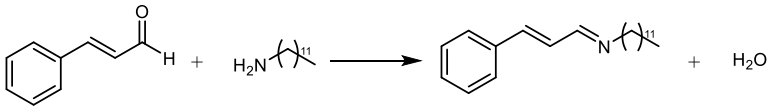


- The nanoreactors for organic synthesis in water


Gel


Aerogel


Powder



98% yield @25°C, 5 min

- Selective deuteration has been achieved (Ru NPs)

- Ag NPs has been achieved on sp^2 carbon allotropes