Consulta Interassociativa Italiana per la Prevenzione

Presentazione del Documento CIIP: punti salienti e problematiche aperte

Carlo Sala

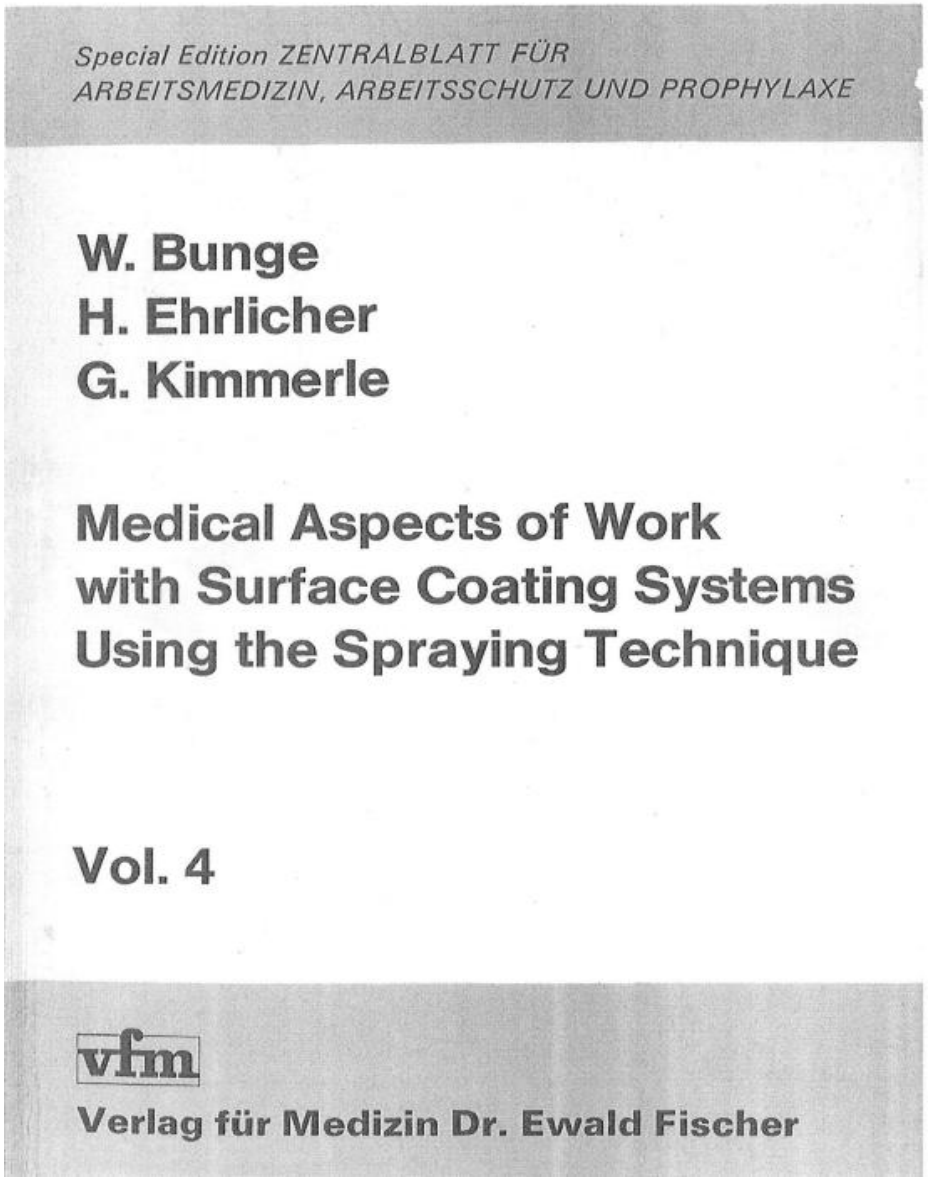
20 marzo 2024

14:00-18:00

Milano

Centro Congressi FAST - Aula Morandi

Identificazione dei rischi della verniciatura a spruzzo di vernici poliuretaniche (1977)



Punti salienti

Obiettivo prioritario : Sostituzione dei diisocianati

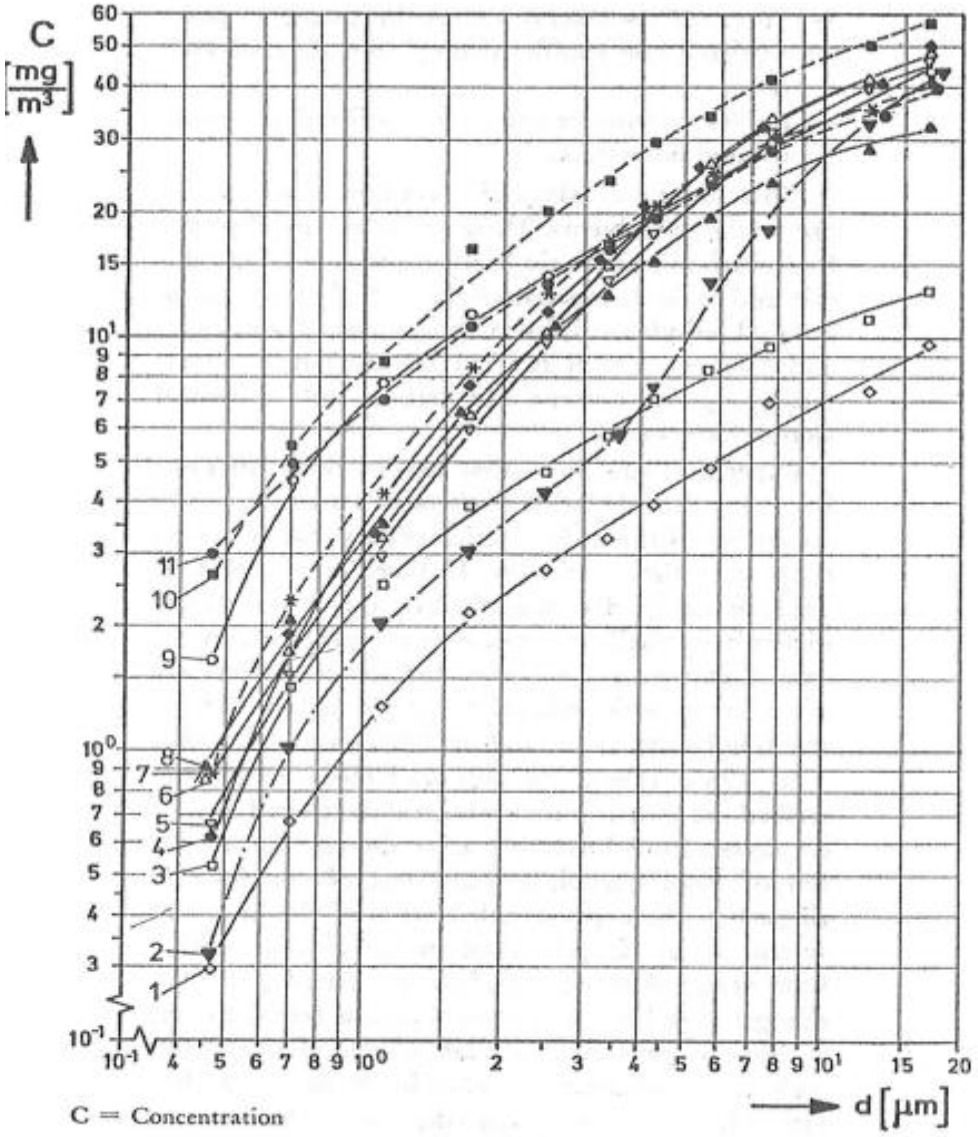
- Eliminazione del fosgene
- Utilizzo di reazioni alternative
- Ricerca di molecole a minore impatto

Obiettivo intermedio: Riduzione al minimo del contenuto di diisocianati

- Formazione di oligomeri
- Formazione di prepolimeri
- Formazione di poliisocianati

Rispetto della restrizione n°74 REACH

Distribuzione della concentrazione e dei diametri delle particelle



Condizioni sperimentali

Table 1: Test Results and Test Conditions of Paint Aerosol Measurements in the Air in Spray Booths

Test Number	Air Pressure (bar) Spraying	Material Pressure (bar)	Vertical Air Speed (m/s) Booth	Aerosol Concentration $< 8\ \mu m$ (mg/m^3)	Curve Number
1	6	2.8		30	9
2	5	1.8	0.35—0.42	9.5	3
3	4.5	2.0		5.1	1
4	5	2.0		30	5
5	5	2.0		34	6
6	6	4.8—3.0		35	7
7	6	4.8—3.0	0.24—0.30	30	11
8	6	4.8—3.0		43	10
9	5.5	4.5	0.28	34	4
10	5.5	2.0	0.35—0.42	25	8
11	7	4.0	0.35—0.42	22	2

HAZARD EVALUATION SYSTEM & INFORMATION SERVICE California Department of Public Health, Occupational Health Branch 850 Marina Bay Parkway, Building P, 3rd Floor, Richmond, CA 94804 HESIS 510-620-5757 • www.cdph.ca.gov/ohb MAY 2014 California Department of Public Health • California Department of Industrial Relations

Worker applying spray-on polyurethane truck bed liner Source: Wikimedia / Creative Commons

Worker applying spray paint to automobile
Source: Auto Prévention (autoprevention.qc.ca)

Spray Polyurethane Foam (SPF)

Worker applying SPF interior insulation

Source: Wikimedia / Creative Commons

Worker applying SPF roofing material

Source: Thomas Gravenstreter

automatizzazione

Contenuto di diisocianati liberi nelle vernici poliuretaniche

Il **Desmodur TH** (Bayer) con un contenuto di TDI libero intorno al 10% è stato ritirato dal mercato nel 1955.

Dopo il 1955 e fino agli anni 70 il valore di TDI libero è stato ridotto progressivamente fino allo 0,7% in 3 modi principali

- Con l'introduzione del **Desmodur L** (Bayer) basato sulla reazione di 3 molecole di TDI con il trimetilolpropano
- Formando poliisocianati in forma di biureto come il trimero dell'HDI **Desmodur N** (Bayer)
- Formando poliisocianati in forma di isocianurati attraverso la trimerizzazione del TDI **Desmodur IL** (Bayer)

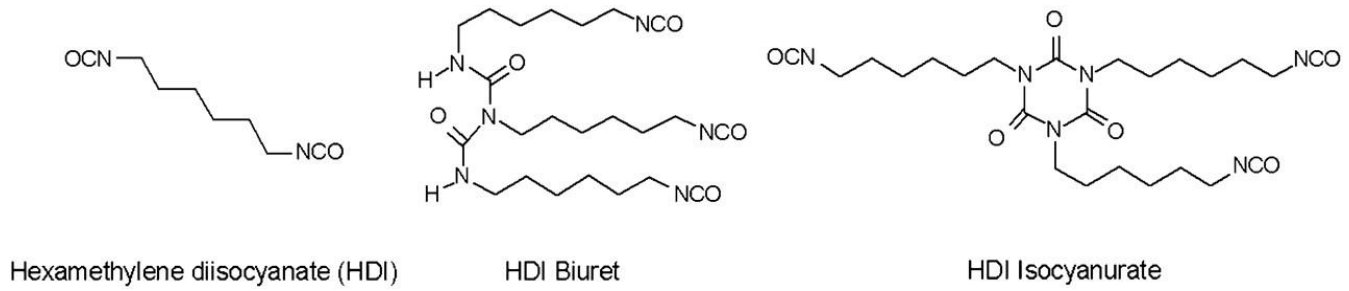


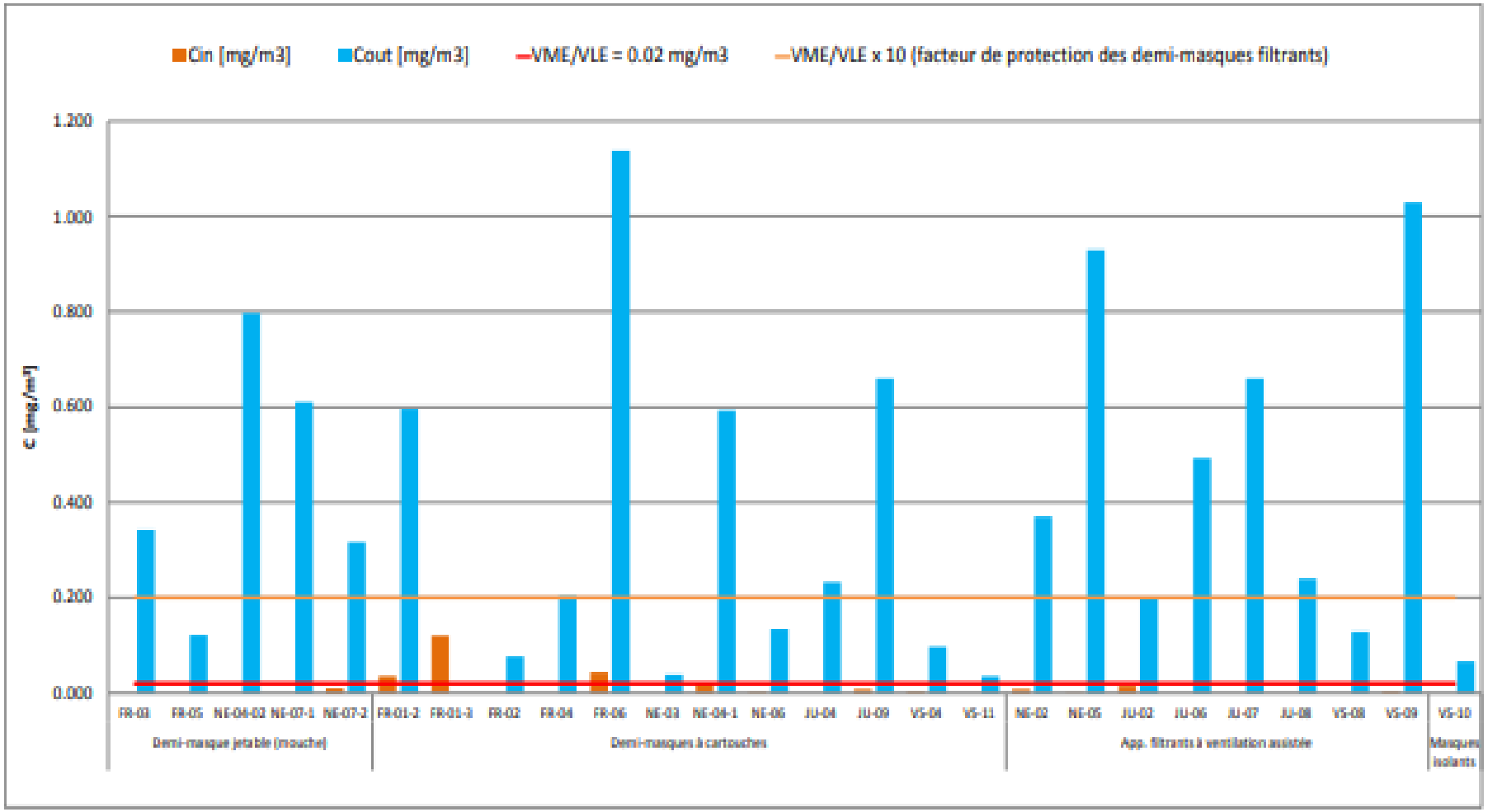
Figure 1. Chemical structures of hexamethylene diisocyanate (HDI) and two HDI oligomers.

Am J Respir Crit Care Med,
https://www.atsjournals.org/doi/abs/10.1164/rccm.200702-215OC

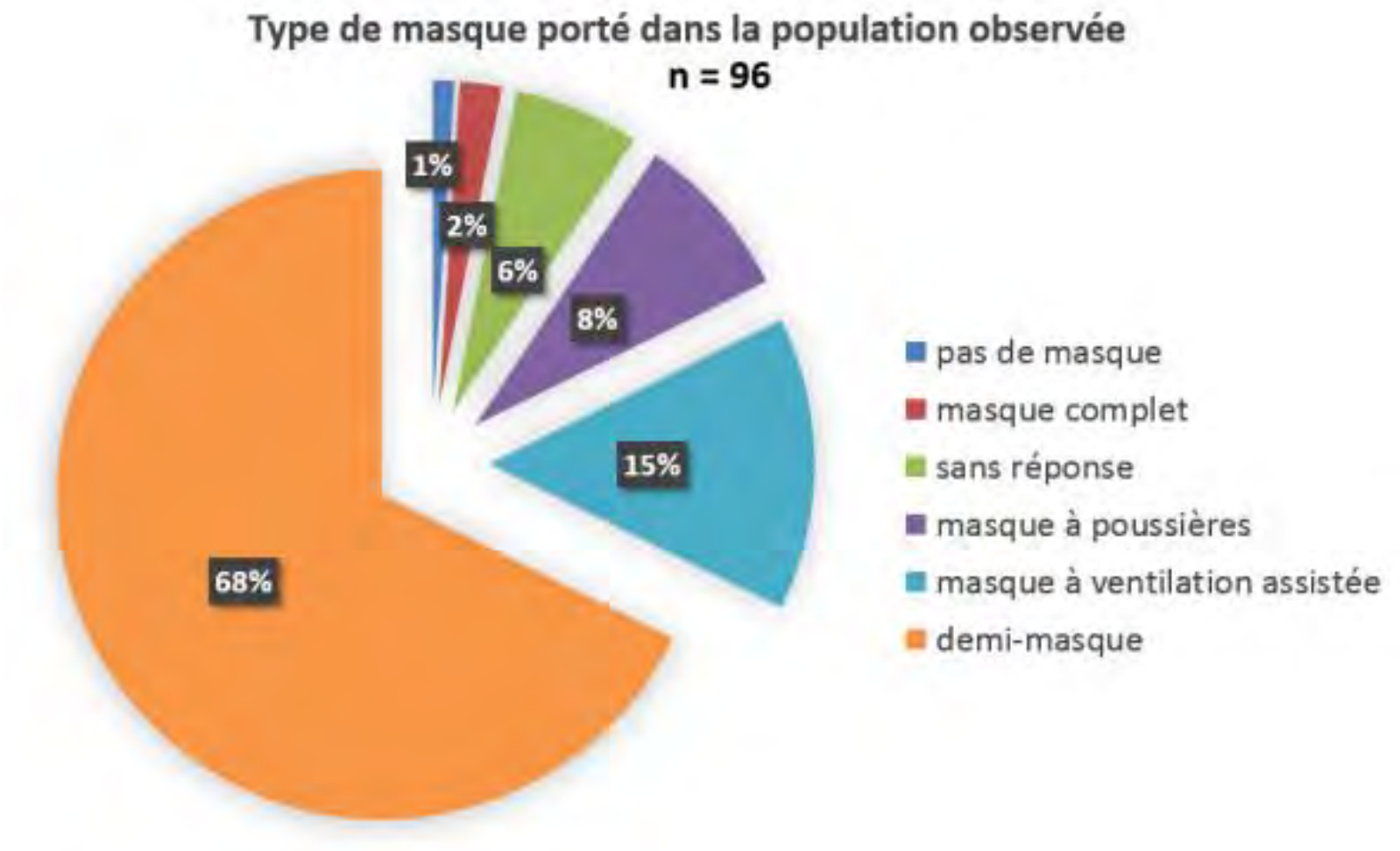
Published in: Anjoeka Pronk; Liesbeth Preller; Monika Raulf-Heimsoth; Irene C. L. Jonkers; Jan-Willem Lammers; Inge M. Wouters; Gert Doekes; Adam V. Wisnewski; Dick Heederik; *Am J Respir Crit Care Med* 2007 1761090-1097.
DOI: 10.1164/rccm.200702-215OC

LABORATOIRE INTERCANTONAL DE SANTE AU TRAVAIL – Une collaboration entre les cantons du Jura, Fribourg, Neuchâtel et Valais 2016 - 2017 Pulvérisation au pistolet de vernis et peintures contenant des isocyanates dans les carrosseries – un état des lieux dans quatre cantons suisses

Concentrazioni di NCO all'esterno e all'interno delle maschere



Tipi di maschere utilizzate



Concentrazioni medie

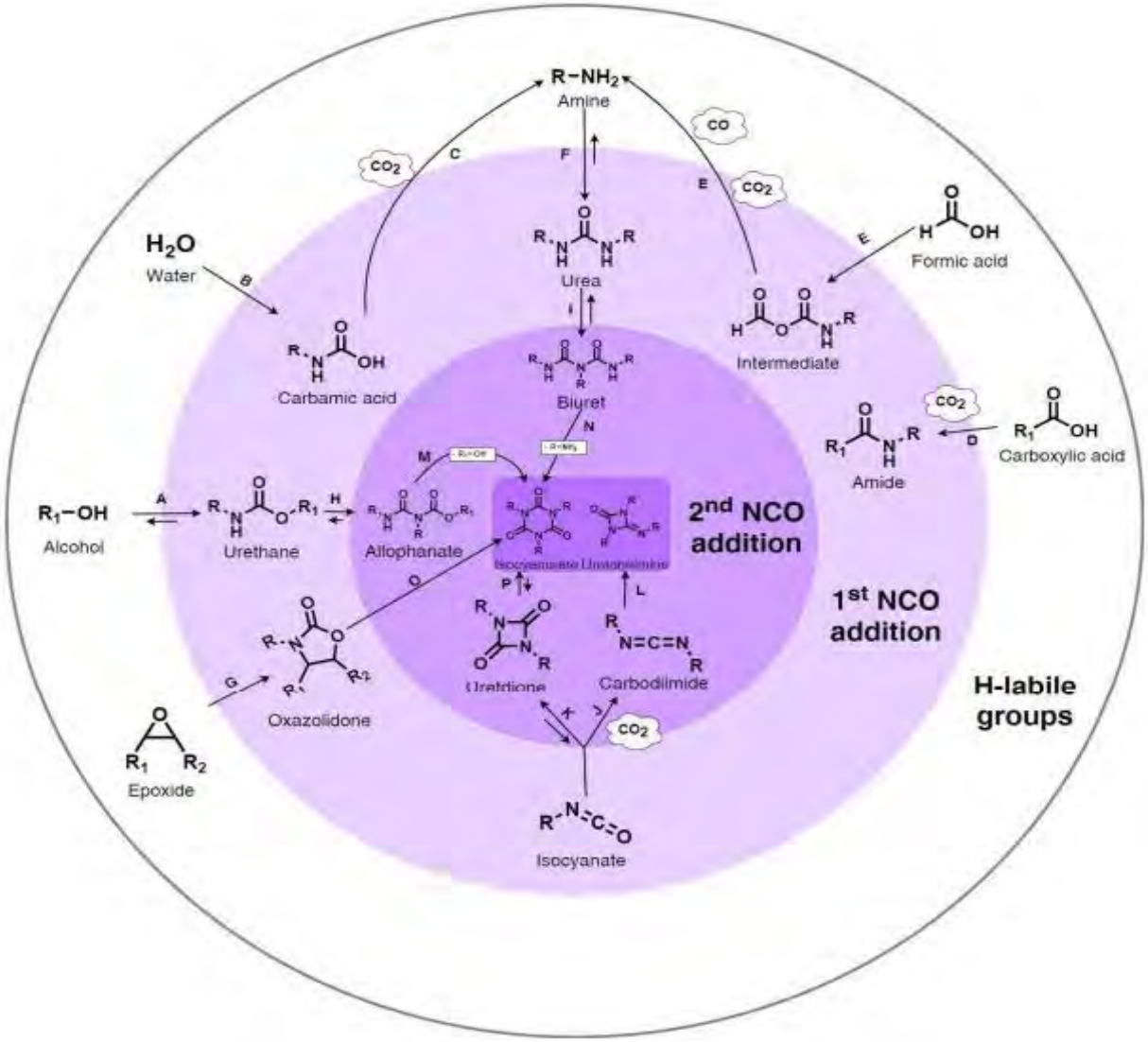
	Concentration moyenne en NCO [µg/m³]		
	Tôlerie	**Cabine**	**Dans le masque**
Nombre de valeurs (N)	27	27	12
Moyenne	0.012	0.341	0.016
Ecart type	0.044	0.329	0.022
Moyenne géométrique	0.003	0.176	0.006

Risultati del monitoraggio biologico

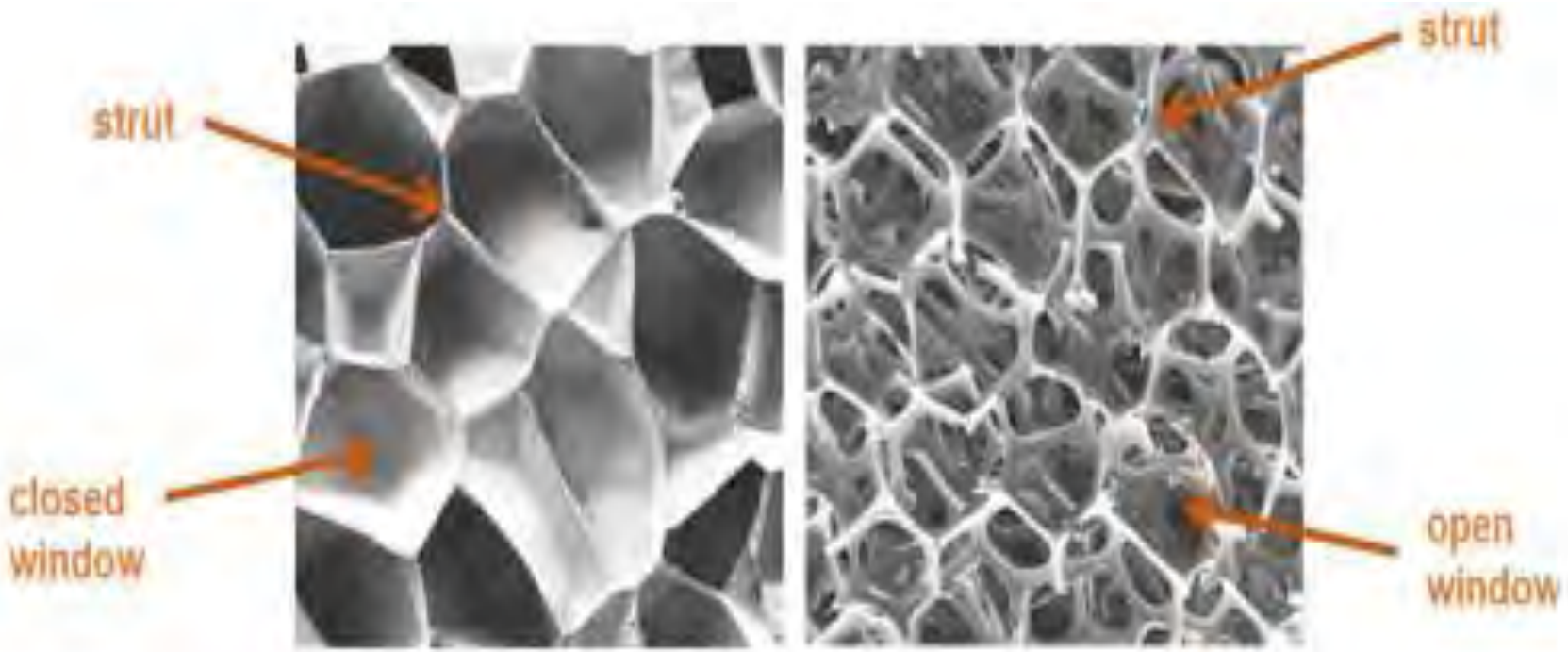
HDA [ug/g créat]	Nombre de personnes		Peinture le jour du prélèvement			
	N	%	OUI	%	NON	%
< 0.7	38	39.2	15	44.1	23	36.5
0.7 – 7.5	44	45.3	16	47.1	28	44.4
7.5 – 15	3	3.1	1	2.9	2	3.2
>15	5	5.2	2	5.9	3	4.8

Versatilità dei diisocianati

Julien Peyrton, Luc Avérous. Structure-properties relationships of cellular materials from biobased polyurethane foams. Materials Science and Engineering: R: Reports, 2021, 145. ffhal-03478358

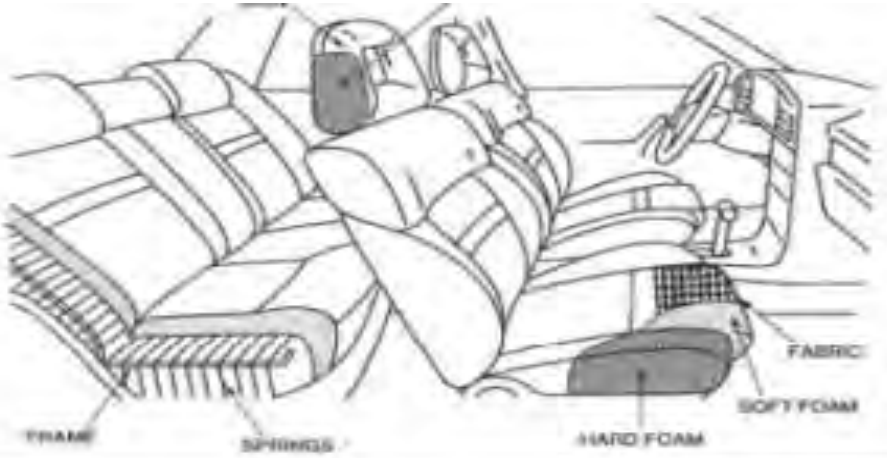


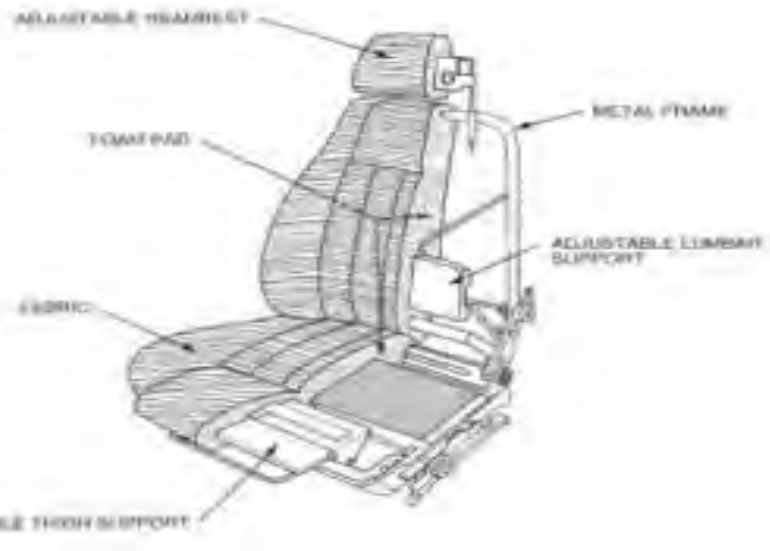
Celle chiuse e celle aperte per schiume rigide e flessibili



Nuove procedure sintetiche sostenibili per la preparazione di strutture poliuretaniche.
Tesi di laurea di Jacopo Bonucci Dipartimento di Chimica Industriale “Toso Montanari”
Università di Bologna. Anno Accademico 2014 - 2015

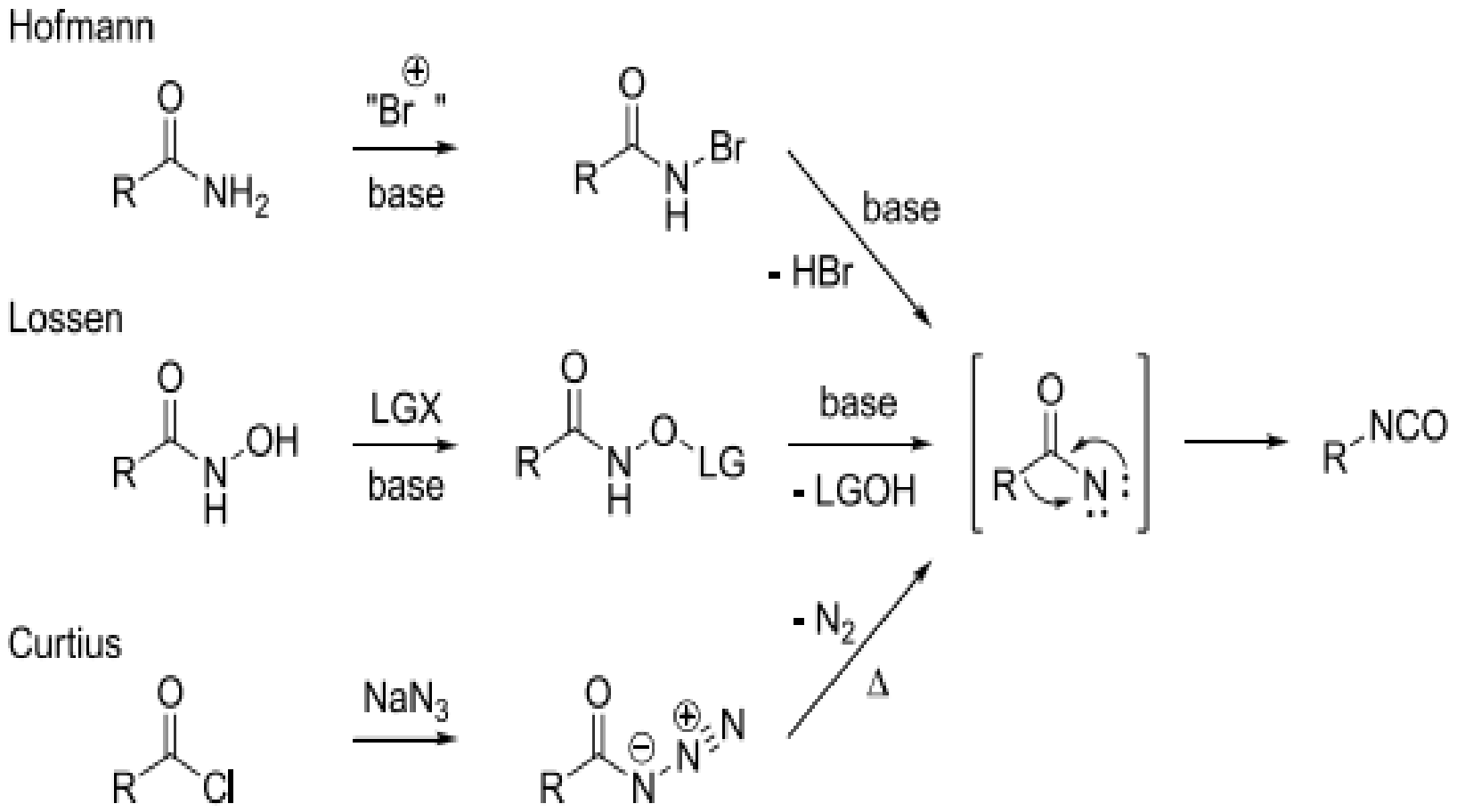
Alcune applicazioni per i sedili dell'auto



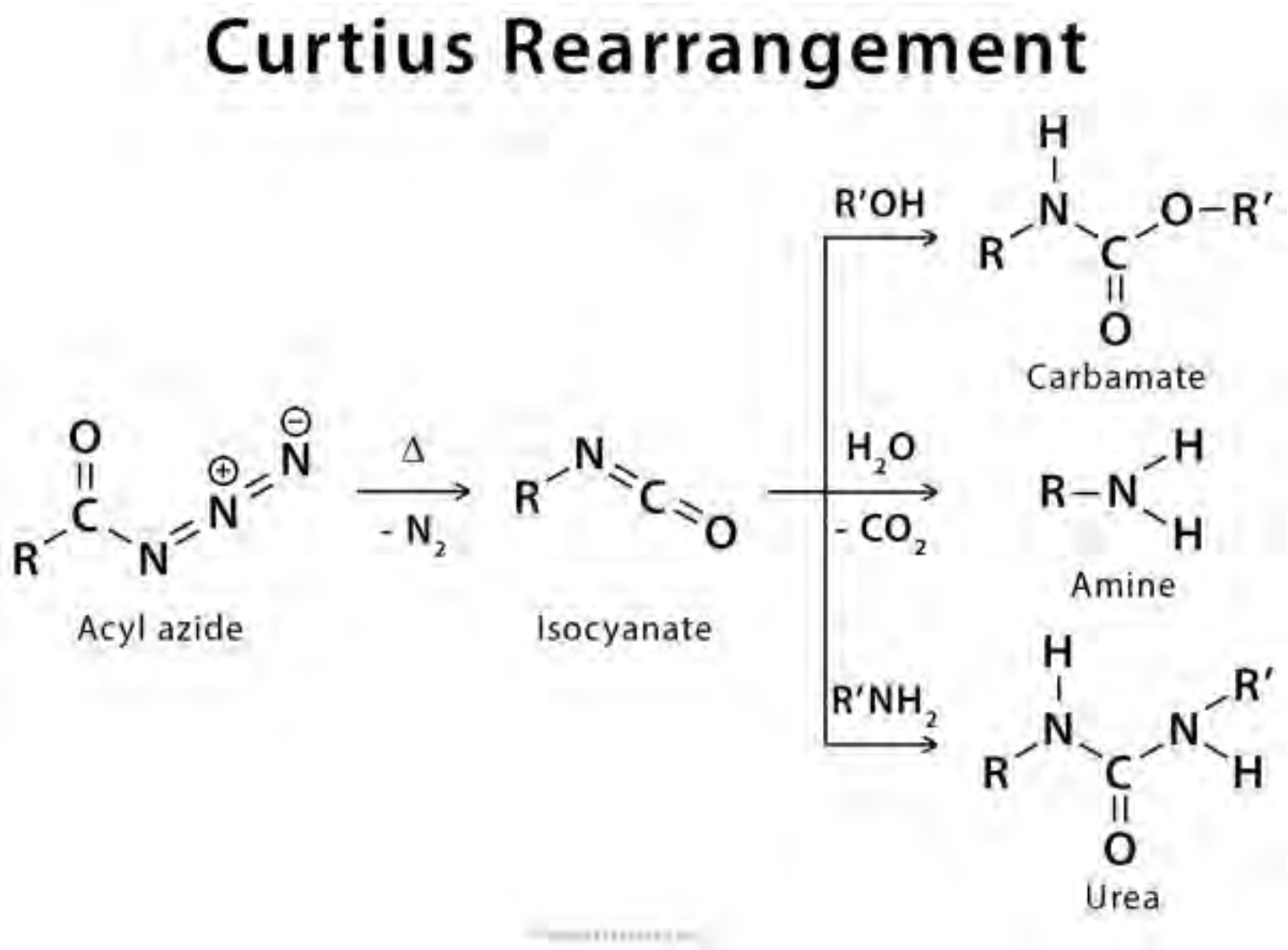


Da documento BASF

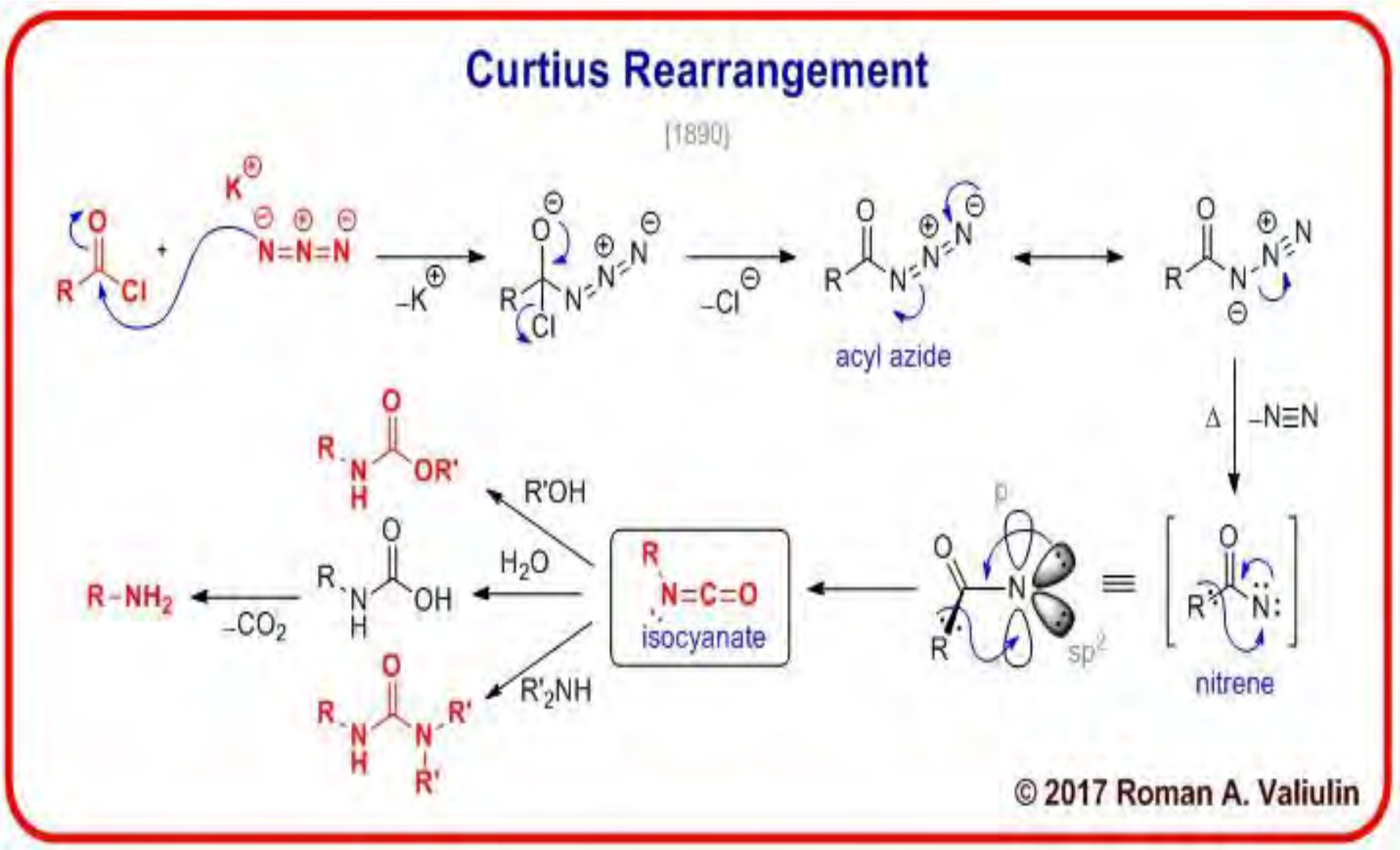
Produzione di poliuretani senza fosgene



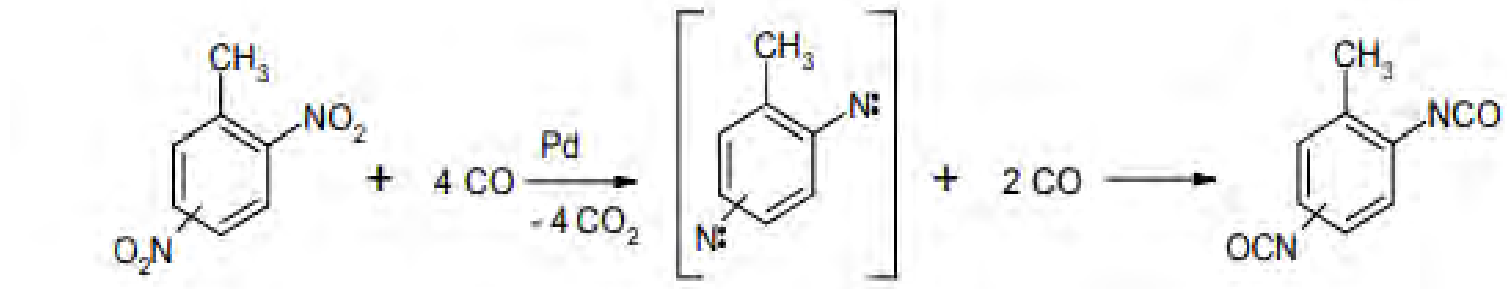
Riarrangiamento di Curtius

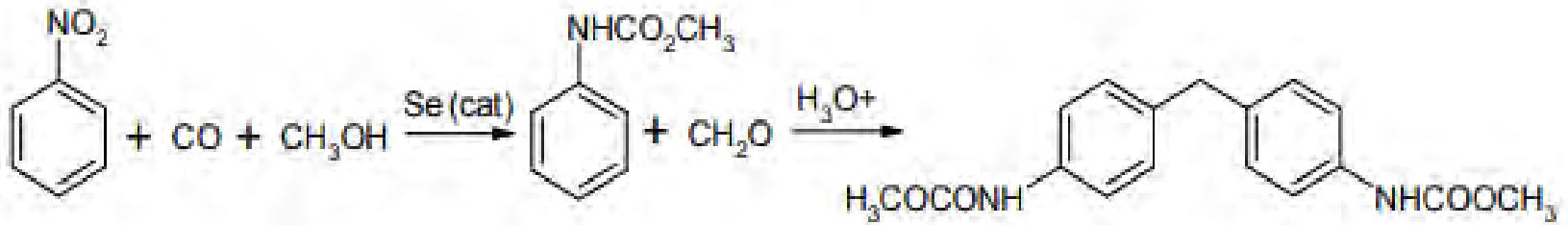


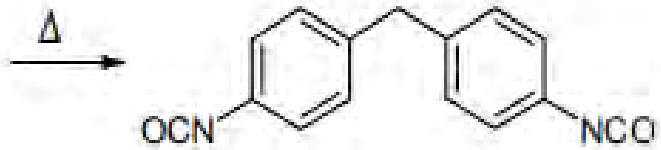
meccanismo


Curtius Rearrangement
[1890]
acyl azide
nitrene
isocyanate
R'OH
H_2O
R'_2NH
$-CO_2$
$R-NH_2$
Δ
$-N\equiv N$
© 2017 Roman A. Valiulin

Studio di Catalisi Termoattivate per Compositi Poliuretanici. tesi di laurea di Valentina Cuoghi presso Università di Bologna Facoltà di Chimica Industriale a.a.2011-2012



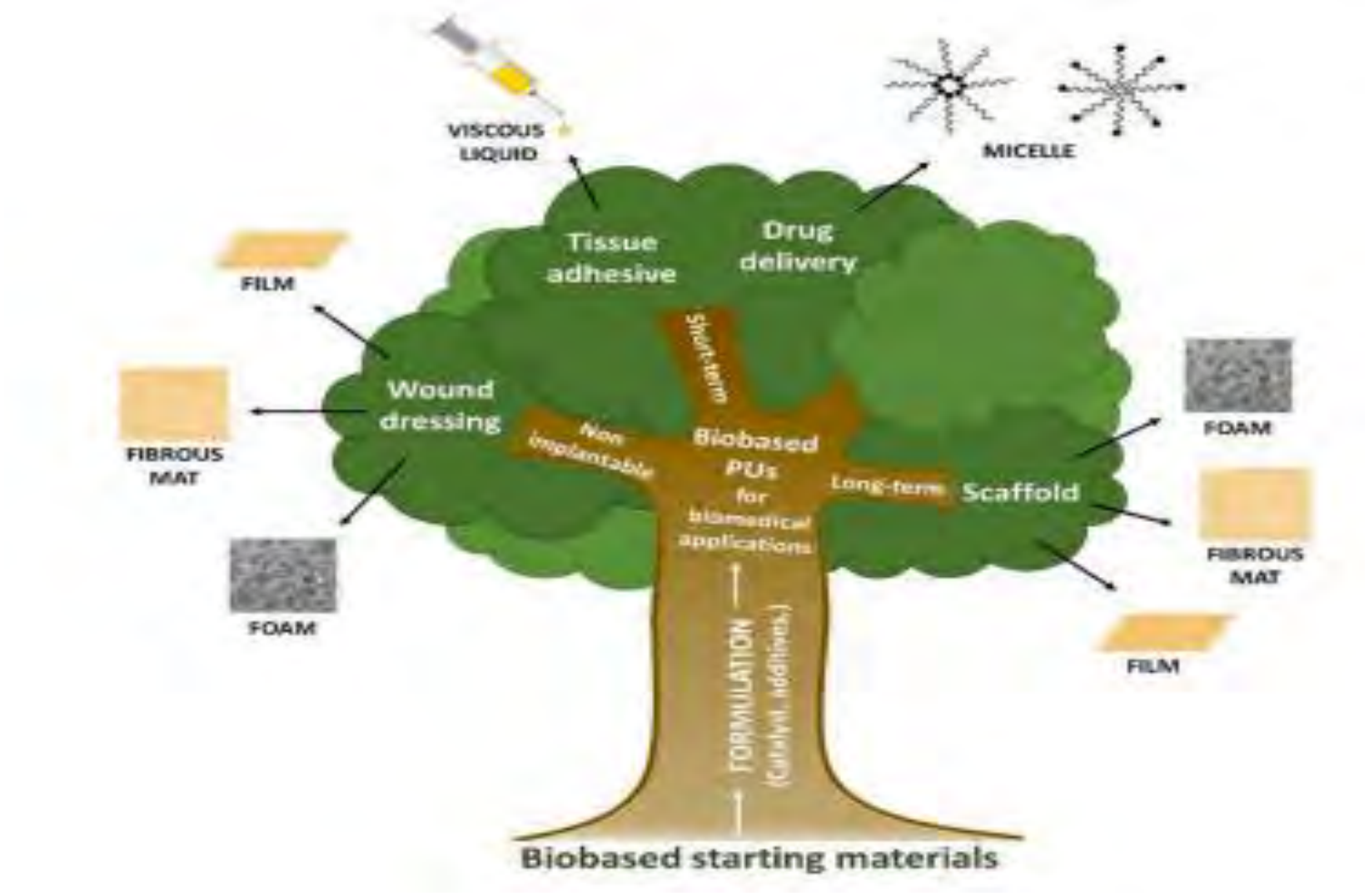




Brevetti per la fosgenazione in fase gassosa tra il 2005 e il 2017

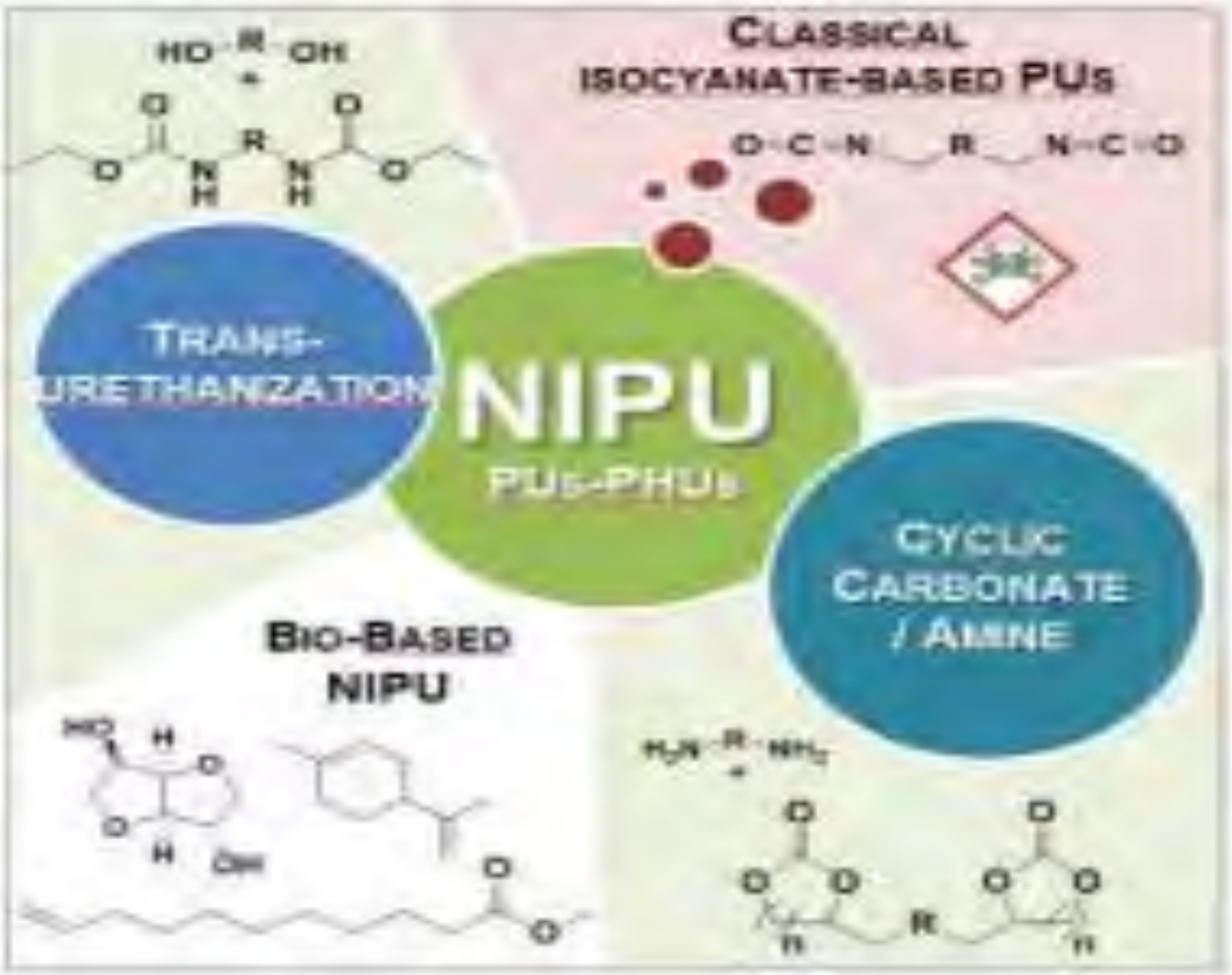
- Wolfert, A.; Muller, C.; Strofer, E.; Weber, M.; Pfeffinger, J.; Knosche, C. ModeratePressure Gas Gas Phase Phosgenation. U.S. Patent 2,005,027,291,0A1, 2005.
- Sanders, J.; Brummer, H.; Laue, J.; Sojka, B.; Eichman, M.; Haverkamp, V. Gas Phase Phosgenation Process. U.S. Patent 2,007,004,323,3A1, 2007.
- Mattke, T.; Olbert, G.; Knoesche, C.; Schelling, H. Method for Producing Diisocyanates by Gas-Phase Phosgenation. U.S. Patent 8,716,517, B2, 2014.
- Knauf, T.; Wolfgang, L.; Friedhelm, S.; Rainer, B.; Wolfgang, T. Process for Operating a Gas Phase Phosgenation Plant. U.S.Patent 2,017,009,638,9A1, 2017

Sophie Wendels, Luc Averous: *Biobased polyurethanes for biomedical applications* .Bioactive Materials 6 (2021) 1083–1106



Isocyanate-free routes to polyurethanes and poly(hydroxy urethane)s

Lise Maisonneuve,1,2 Océane Lamarzelle,1,2 Estelle Rix, 1,2 Etienne Grau1,2 and Henri Cramail1,2
1 Univ. Bordeaux, LCPO, UMR 5629, F-33600, Pessac, France ; 2 CNRS, LCPO, UMR 5629, F-33600, Pessac, France *cramail@enscbp.fr



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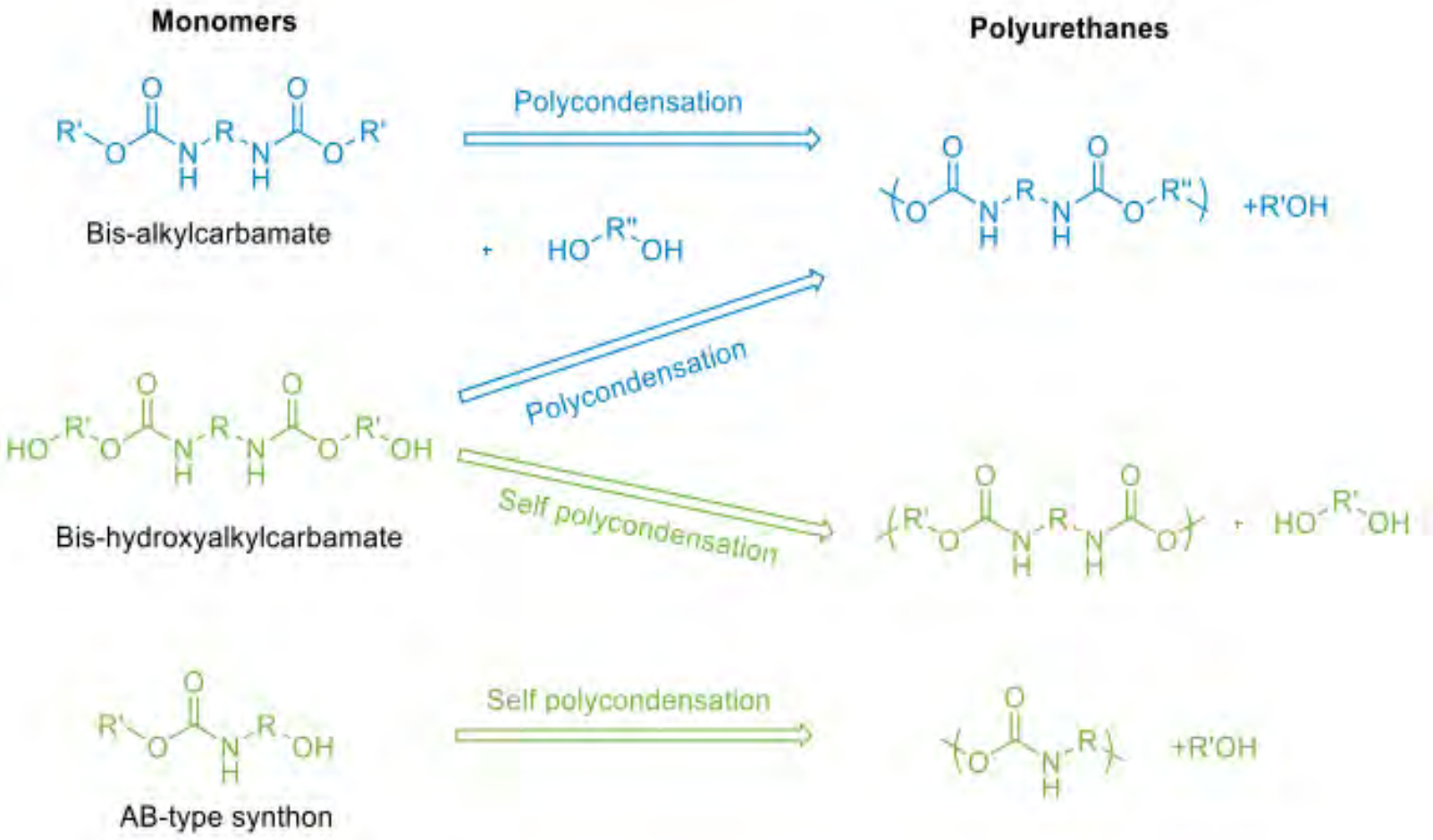
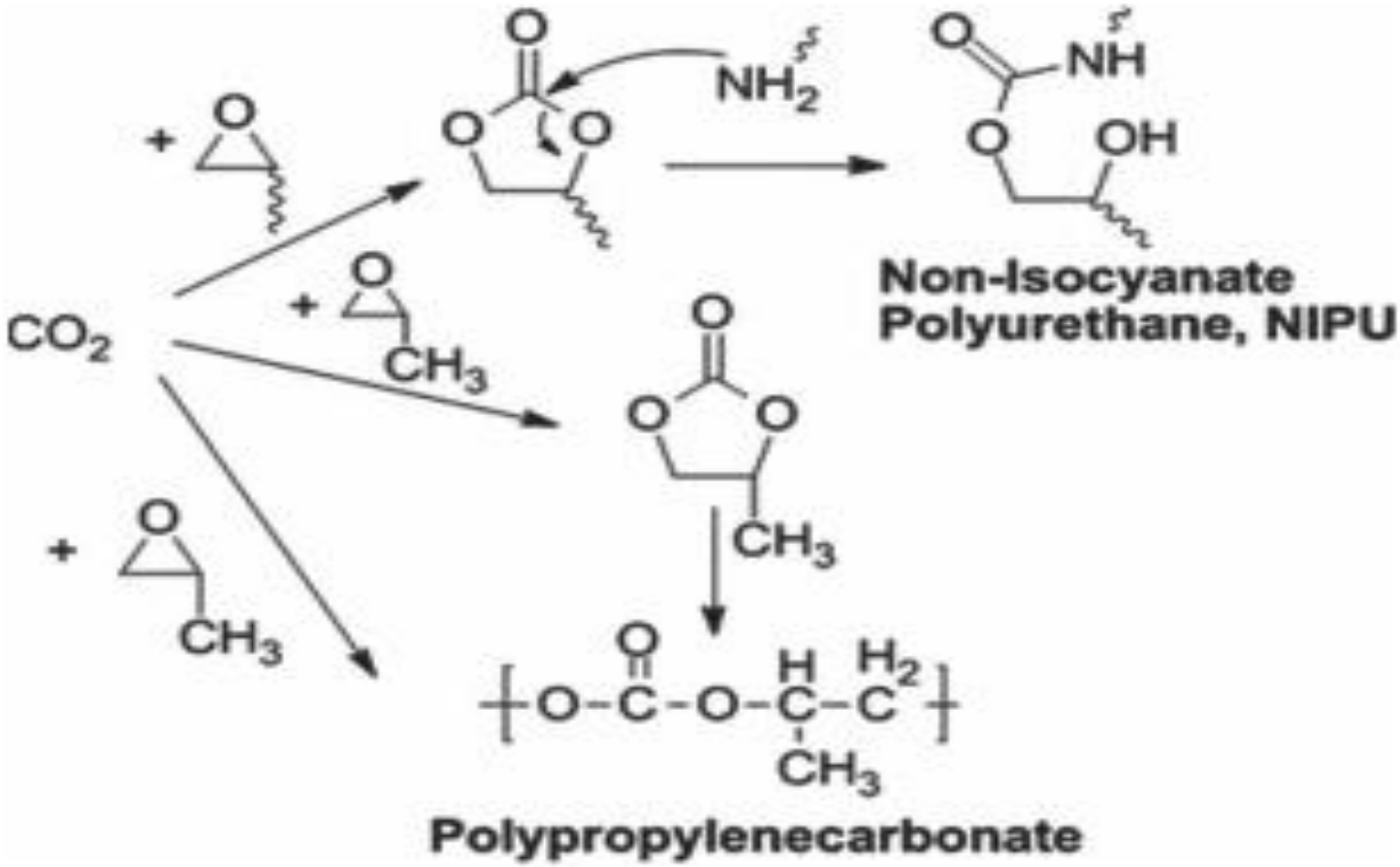
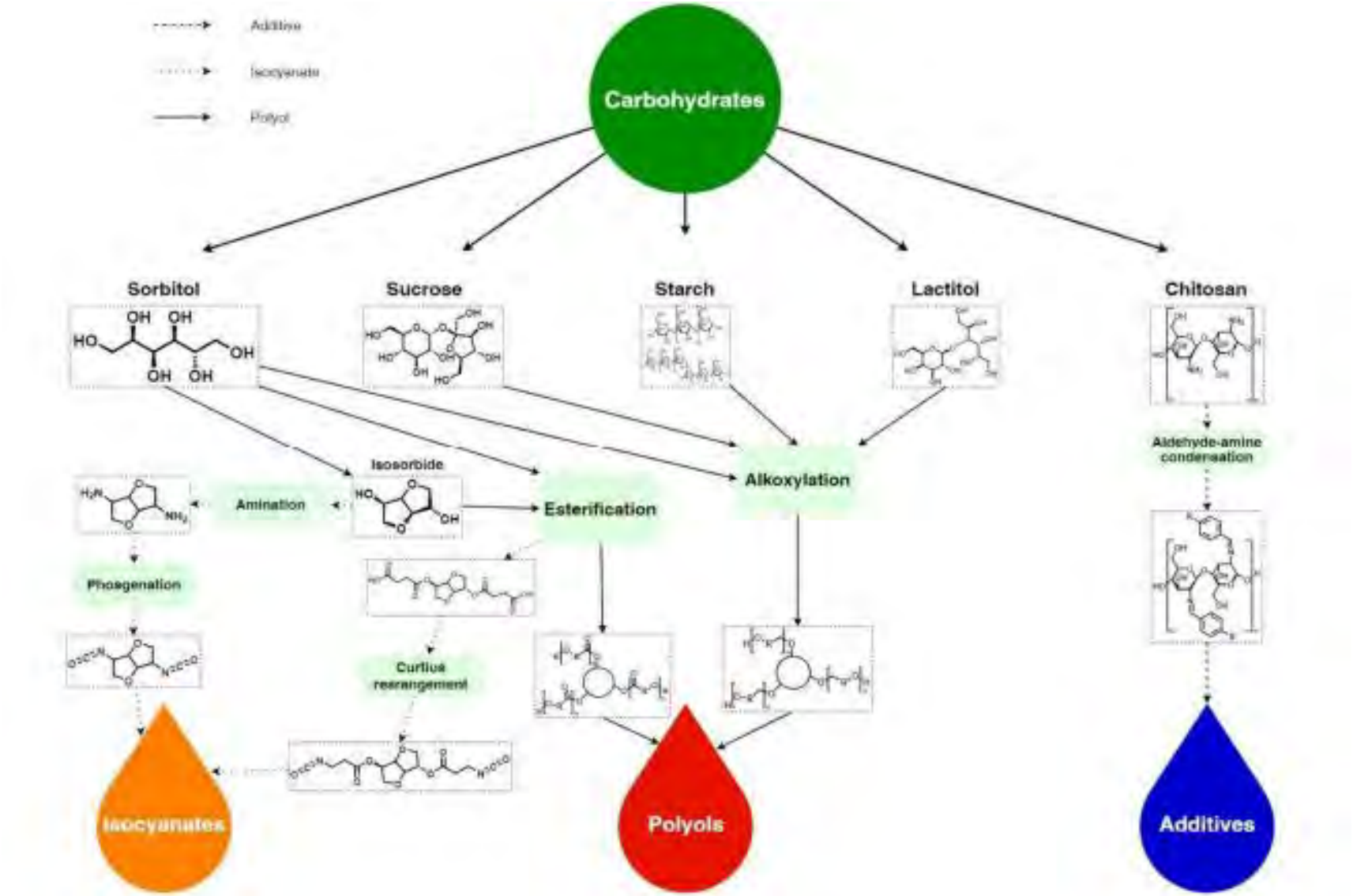


R. Mülhaupt **Green Polymer Chemistry and Bio-based Plastics: Dreams and Reality**
Macromol. Chem. Phys. 2013, 214, 159−174 © 2013 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim



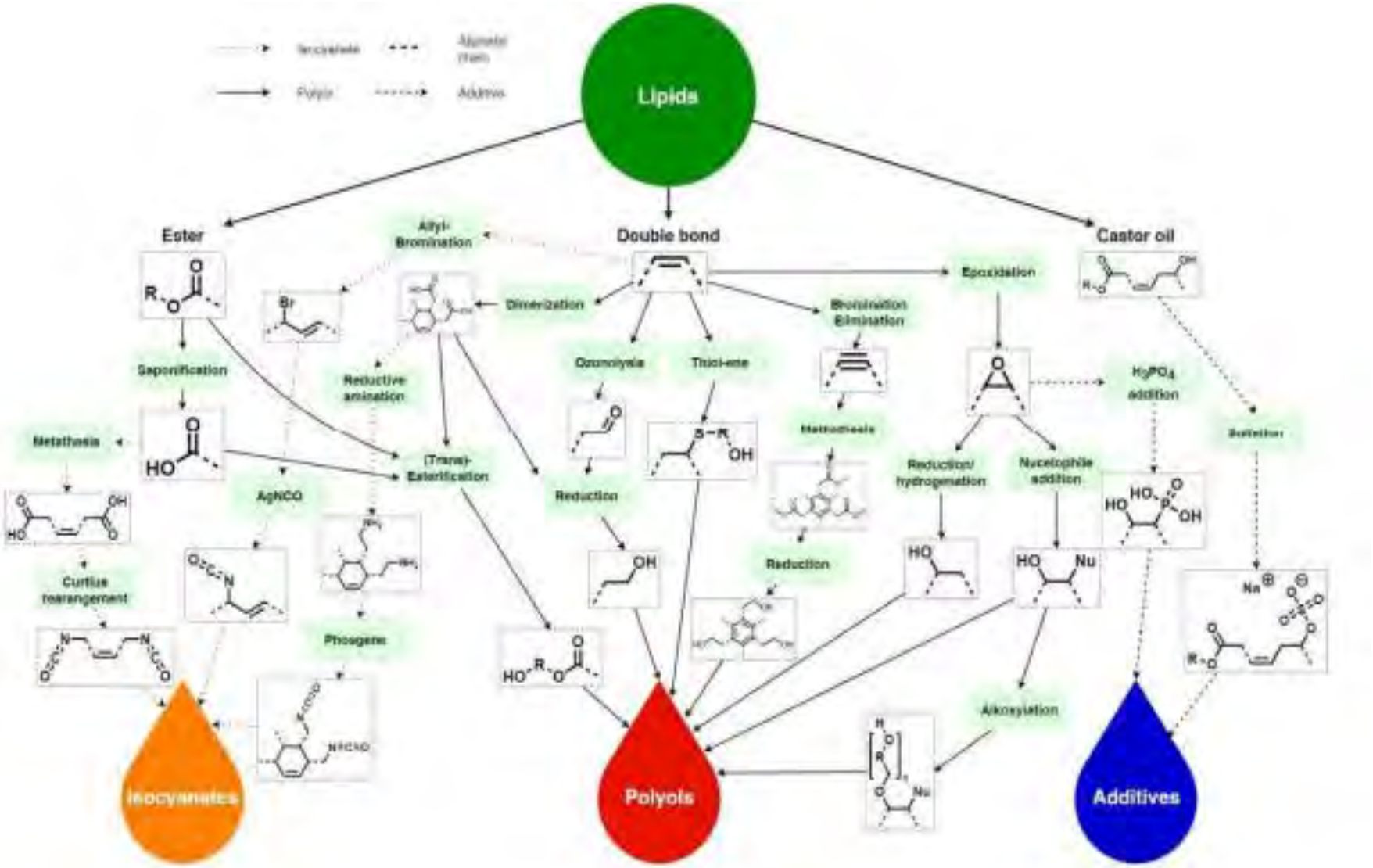
biobased diisocyanates synthesis

Julien Peyrton, Luc Avérous. Structure-properties relationships of cellular materials from biobased polyurethane foams. Materials Science and Engineering: R: Reports, 2021, 145. ffhal-03478358



main oleochemistry pathways for the elaboration of different PUF components

Julien Peyrton, Luc Avérous. Structure-properties relationships of cellular materials from biobased polyurethane foams. Materials Science and Engineering: R: Reports, 2021, 145. ffhal-03478358



150 °anniversario attività Bayer

Angewandte Reviews

H.-W. Engels et al.

DOI: 10.1002/anie.201302766

Polyurethanes

Polyurethanes: Versatile Materials and Sustainable Problem Solvers for Today's Challenges

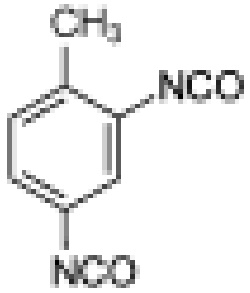
Hans-Wilhelm Engels, Hans-Georg Pirkl, Reinhard Albers, Rolf W. Albach, Jens Krause, Andreas Hoffmann, Holger Casselmann, and Jeff Dormish*

Keywords:
adhesives · coatings · elastomers · flexible foams · rigid foams

Dedicated to the Bayer company on the occasion of its 150th anniversary

Diisocianati rilevanti dal punto di vista industriale

H.-W. Engels et al. Polyurethanes: Versatile Materials and Sustainable Problem Solvers for Todays Challenges. Angew. Chem. Int. Ed. 2013, 52, 9422 – 9441



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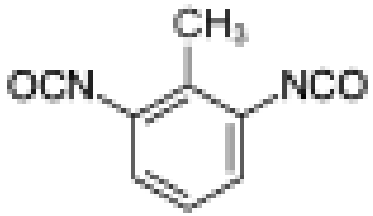

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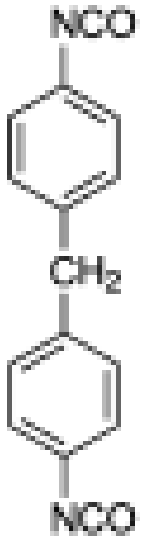

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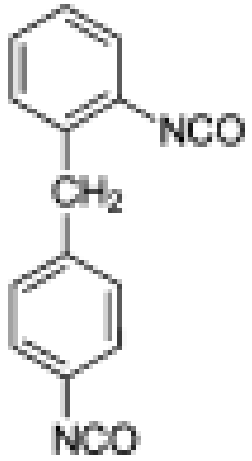

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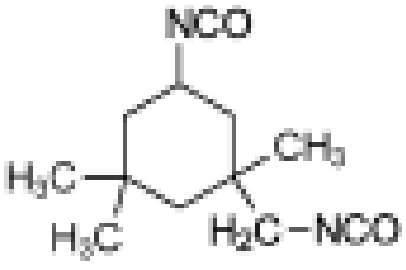

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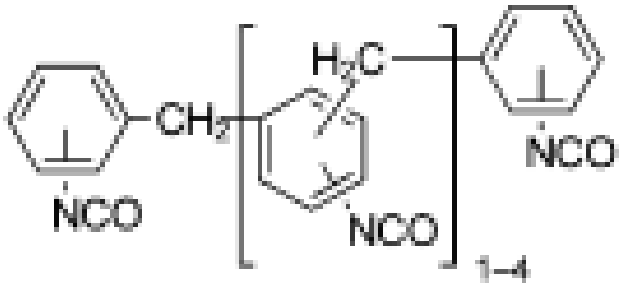
Hans-Wilhelm Engels et al. Polyurethanes: Versatile Materials and Sustainable Problem Solvers for Todays Challenges. Angew. Chem. Int. Ed. 2013, 52, 9422 – 9441



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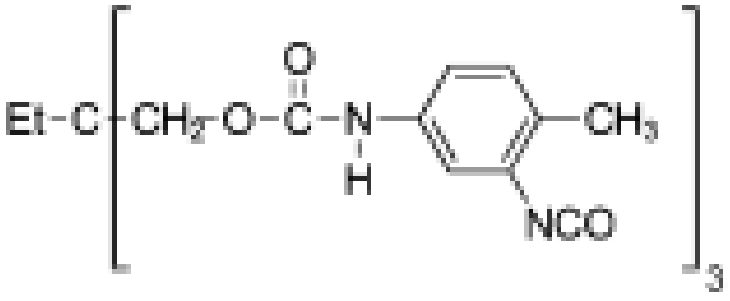

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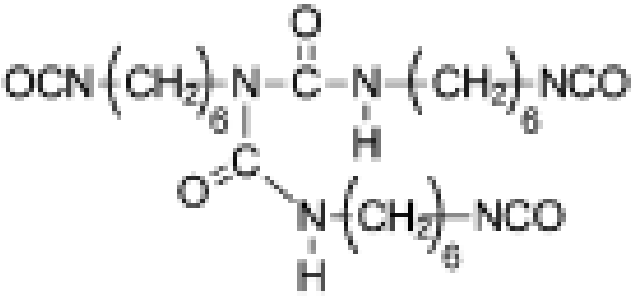

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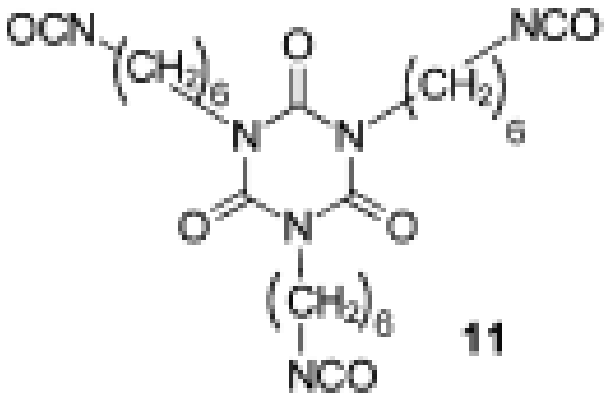


11

Selected diisocyanates in the European market (Source: European Chemicals Agency, http://echa.europa.eu/)

Abbrev.	Chemical Name	CAS No.	Manufacture and/or import in the European Economic Area (amount, tonnes per year)	Harmonised health hazard Classification under the CLP regulation (Annex VI of Regulation (EC) No 1272/2008)
MDI	4,4'-methylenediphenyl diisocyanate + other isomers	101-68-8	100 000 – 1 000 000	Skin Irrit. 2, Eye Irrit. 2, Skin Sens. 1, Acute Tox. 4 *, STOT SE 3, Resp. Sens. 1, Carc. 2, STOT RE 2 *
TDI	4-methyl-m-phenylene diisocyanate + other isomers	584-84-9	100 000 – 1 000 000	Skin Irrit. 2, Eye Irrit. 2, Skin Sens. 1, Acute Tox. 2 *, STOT SE 3, Resp. Sens. 1, Carc. 2
HDI	hexamethylene diisocyanate	822-06-0	10 000 – 100 000	Skin Irrit. 2, Eye Irrit. 2, Skin Sens. 1, Acute Tox. 3 *, STOT SE 3, Resp. Sens. 1
IPDI	3-isocyanatomethyl-3,5,5-trimethylcyclohexyl isocyanate + other isomers	4098-71-9	10 000 – 100 000	Skin Irrit. 2, Eye Irrit. 2, Skin Sens. 1, Acute Tox. 3 *, STOT SE 3, Resp. Sens. 1
HDMI	4,4'-methylenedicyclohexyl diisocyanate	5124-30-1	10 000 – 100 000	Skin Irrit. 2, Eye Irrit. 2, Skin Sens. 1, Acute Tox. 3 *, STOT SE 3, Resp. Sens. 1
NDI	1,5-naphthylene diisocyanate	3173-72-6	1000 – 10 000	Skin Irrit. 2, Eye Irrit. 2, Acute Tox. 4 *, STOT SE 3, Resp. Sens. 1 + Addition of Skin Sens 1A and modification of Acute Tox 4 * to Acute Tox 2 under evaluation
XDI	1,3-bis(isocyanatomethyl)benzene	3634-83-1	1000 – 10 000	n/a Skin Sens. 1A and Resp. Sens. 1 under evaluation
TMXDI	1,3-bis(1-isocyanato-1-methylethyl)benzene	2778-42-9	100 – 1000	n/a Skin Sens. 1A and Resp. Sens. 1 under evaluation
TRIDI	2,4,6-triisopropyl-m-phenylene diisocyanate	2162-73-4	100 – 1000	n/a Skin Sens. 1 and Resp. Sens. 1 under evaluation
TODI	3,3'-dimethylbiphenyl-4,4'-diyl diisocyanate	91-97-4	10 – 100	n/a Skin Sens. 1A and Resp. Sens. 1 under evaluation

** Indicates that manufacturers or importers must apply at least this minimum classification, but must classify in a more severe hazard category in the event that further information is available which shows that the hazard(s) meet the criteria for classification in the more severe category (See Annex VI, Section 1.2.1 of the CLP Regulation)*

Uso di CO_2 liquida come agente espandente

Hans-Wilhelm Engels et al. Polyurethanes: Versatile Materials and Sustainable Problem Solvers for Todays Challenges. Angew. Chem. Int. Ed. 2013, 52, 9422 – 9441

Impianto pilota

Rigid polyurethanes foaming with CO2 as physical blowing agent

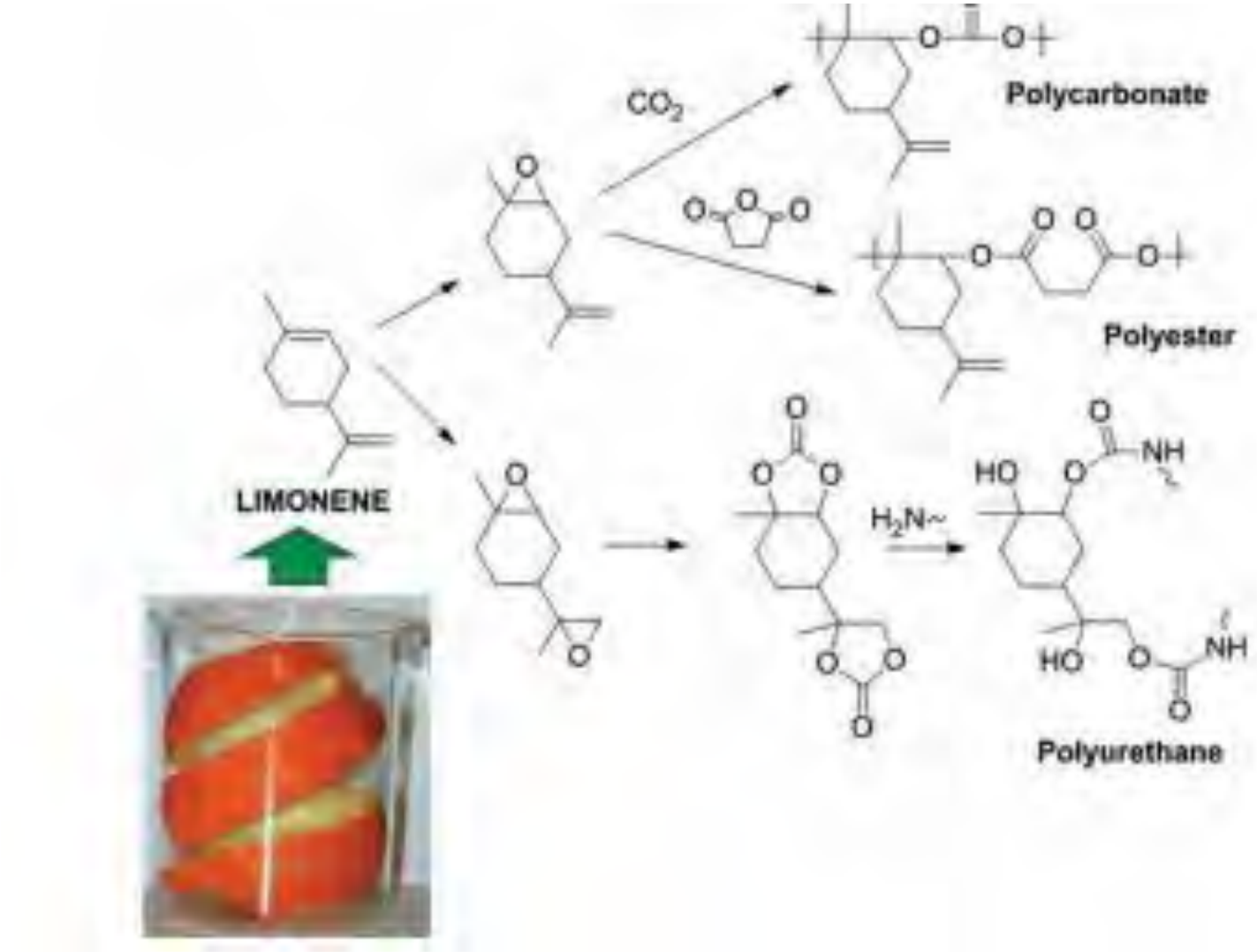
Maria Rosaria Di Caprio PhD in Industrial Product and Process Engineering (XXX Cycle) Department of Chemical, Material and Industrial Production Engineering University of Naples FEDERICO II

PON Ricerca e Innovazione 2014- 2020
Unione Europea
Ministero dell'Istruzione, dell'Università e della Ricerca
Dottorato di Ricerca in Ingegneria dei Prodotti e dei Processi Industriali

Tubi per il trasferimento di fluidi, coibentati con poliuretani rigidi

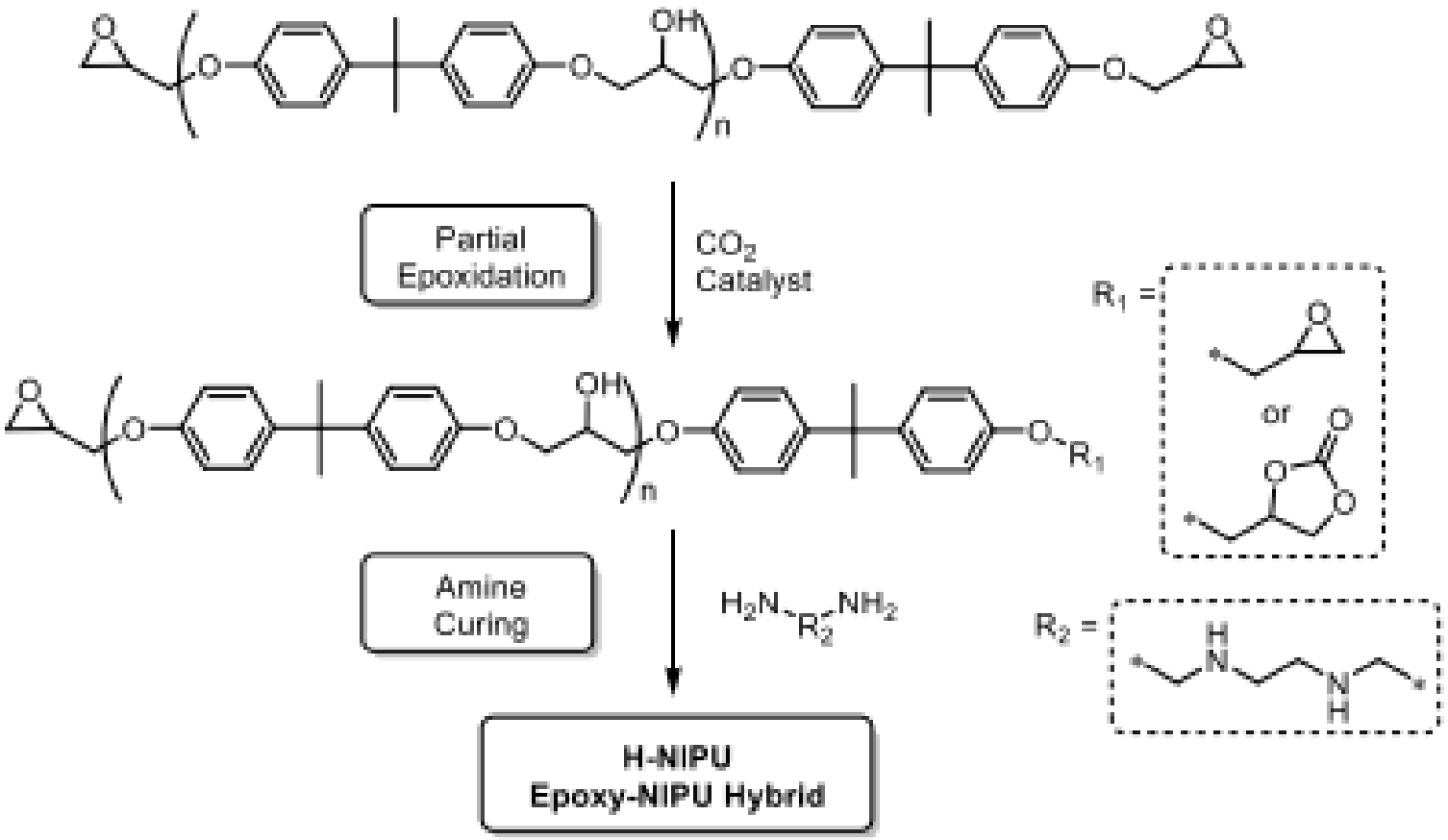
Hans-Wilhelm Engels et al. Polyurethanes: *Versatile Materials and Sustainable Problem Solvers for Todays Challenges.* Angew. Chem. Int. Ed. 2013, 52, 9422 – 9441

Rolf Mülhaupt. Green Polymer Chemistry and Bio-based Plastics: Dreams and Reality. Macromol. Chem. Phys. 2013, 214, 159−174



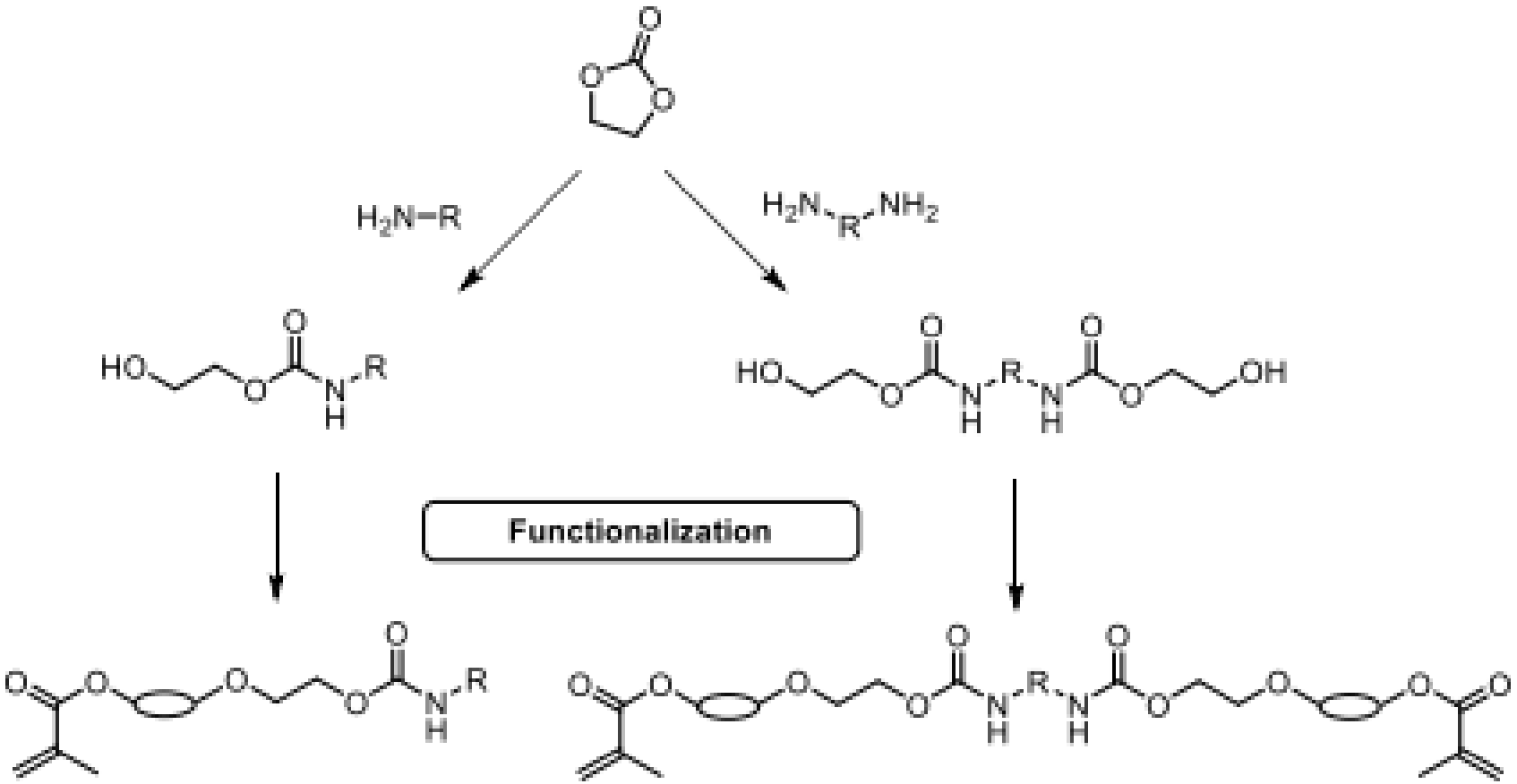
Hybrid-NIPUs (Hybrid – Non Isocyanate Polyurethanes)

tesi di dottorato di Boris Bizet Université de Bordeaux- École Doctorale des Sciences Chimiques-2020



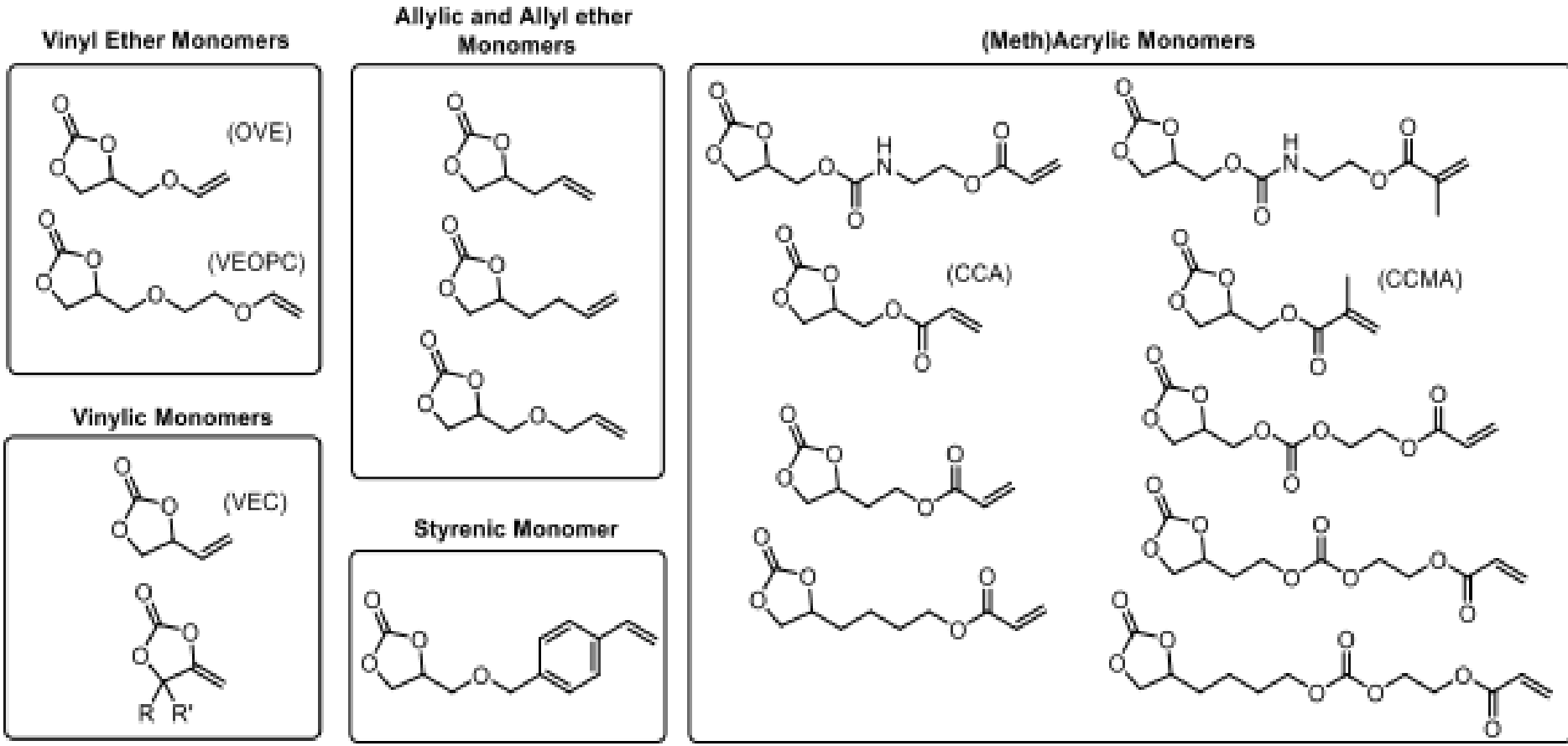
(Hydroxy)urethane methacrylates – (H)UMAs

tesi di dottorato di Boris Bizet Université de Bordeaux- École Doctorale des Sciences Chimiques-2020



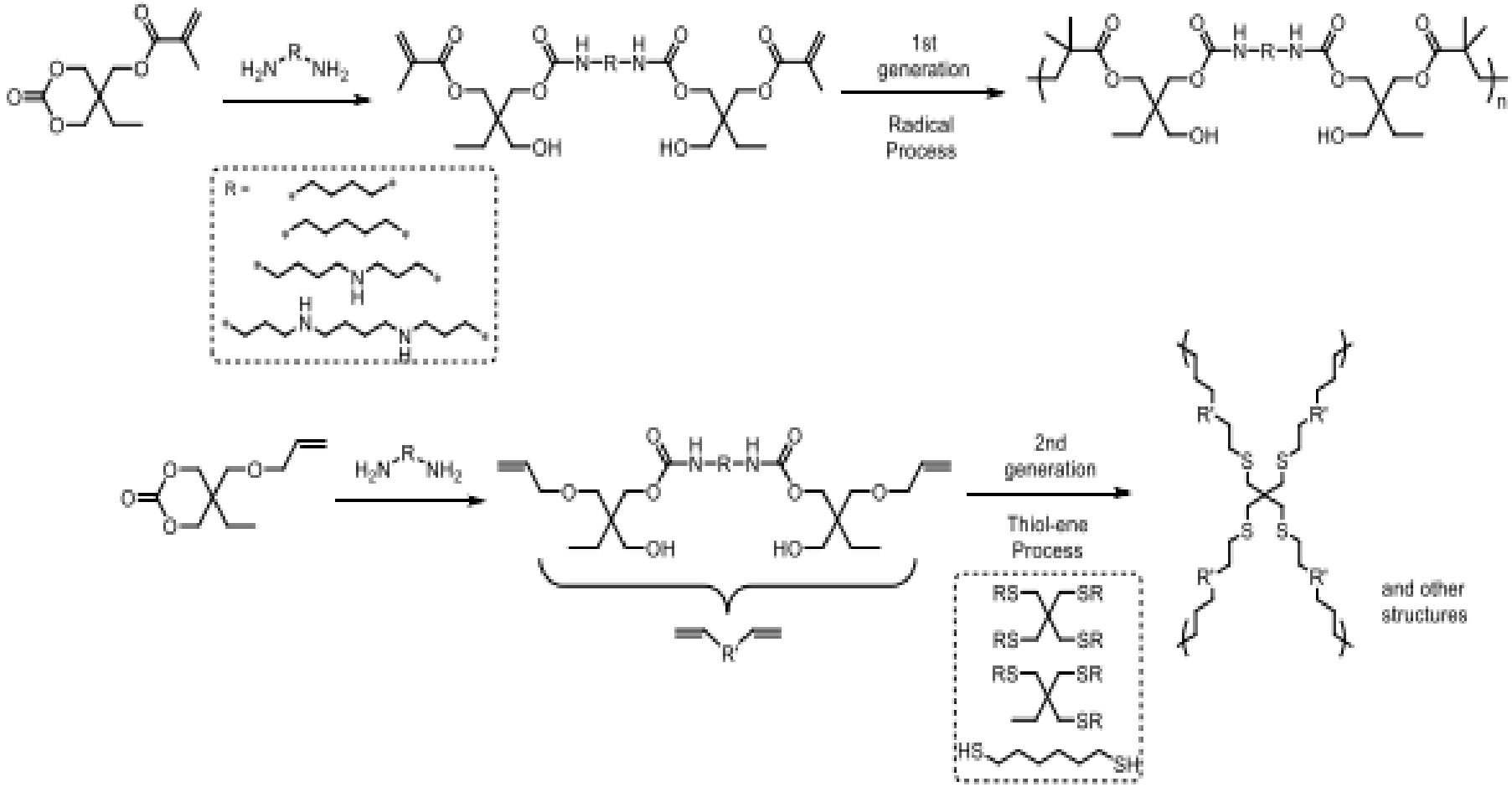
Radical reactive cyclic carbonates

tesi di dottorato di Boris Bizet Université de Bordeaux- École Doctorale des Sciences Chimiques-2020



3D-printable HUMAs

tesi di dottorato di Boris Bizet Université de Bordeaux- École Doctorale des Sciences Chimiques-2020



Stampante 3D per poliuretani modificati

Materials and Design 179 (2019) 107905

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

3D printed cork/polyurethane composite foams

N. Gama [a,*], A. Ferreira [b], A. Barros-Timmons [a]

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[b] CICECO - Aveiro Institute of Materials and Escola Superior de Tecnologia e Gestão de Águeda - Rua Comandante Pinho e Freitas, no 28, 3750 – 127 Águeda, Portugal

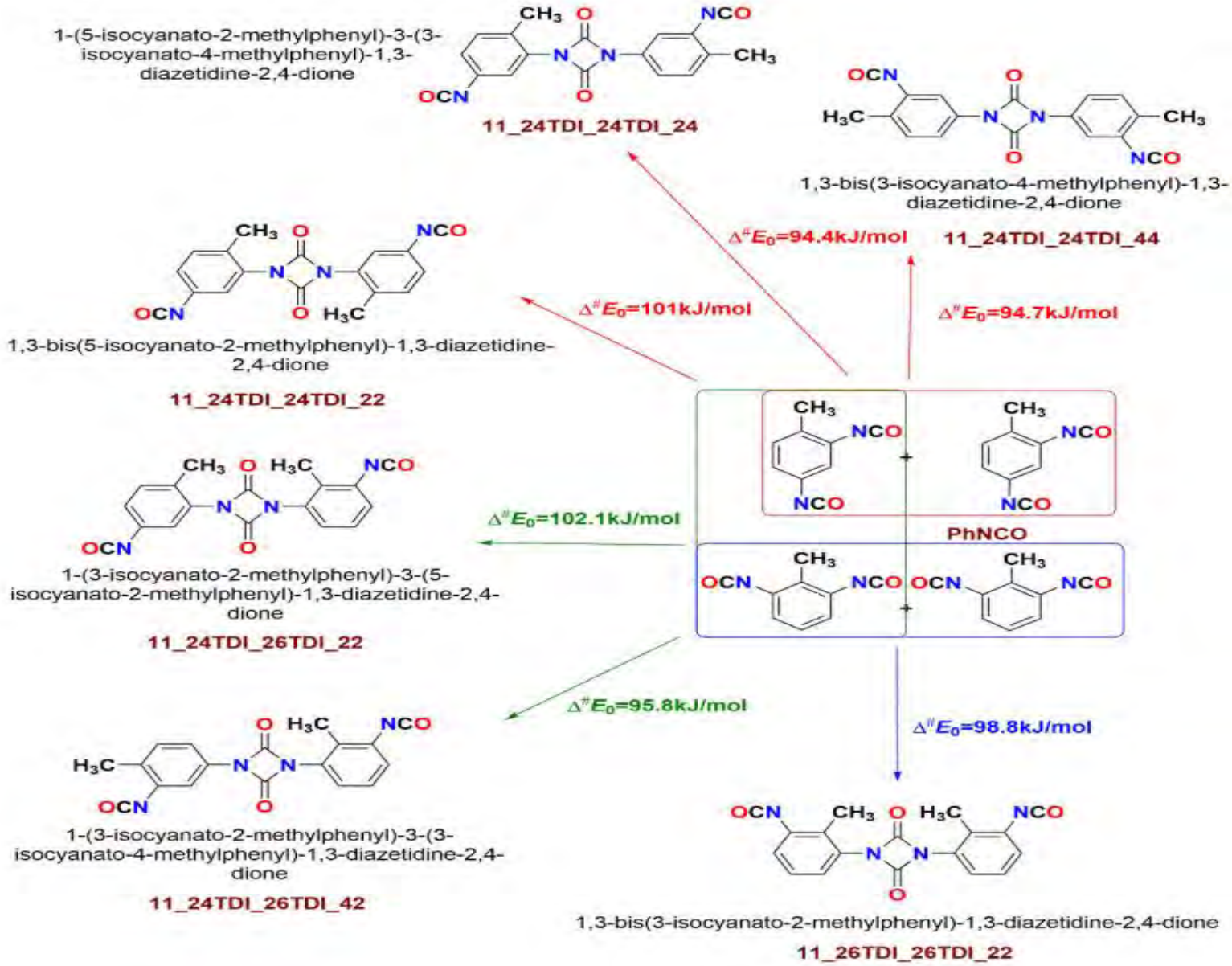
HIGHLIGHTS

- Polyurethane foams were produced using 3D printing technology.
- Cork was used to enhance the properties of the 3D printed composite foams.
- The 3D printed foams have potential to be used in applications beyond thermal insulation.

GRAPHICAL ABSTRACT

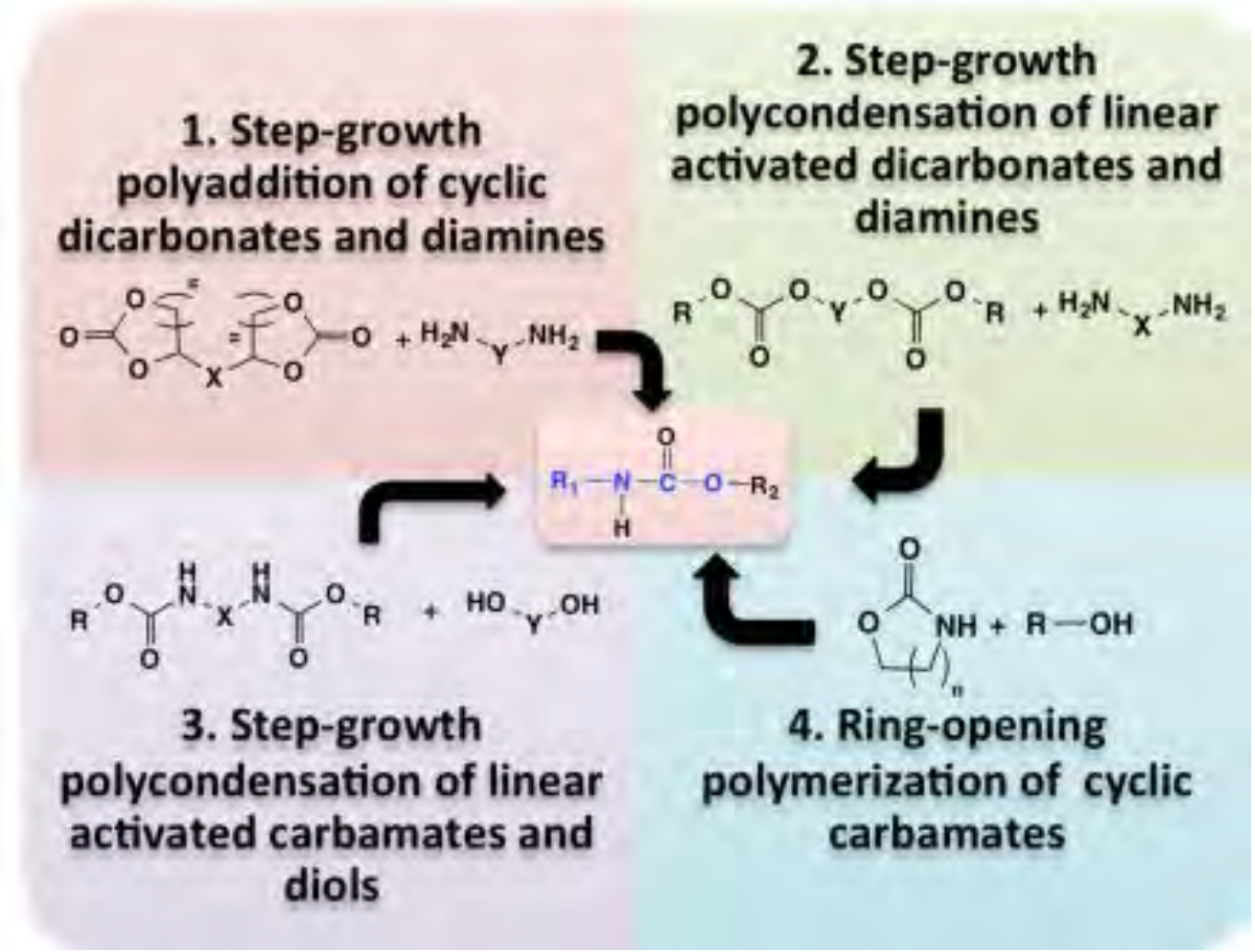
Ciclodimerizzazione del TDI

Ravikumar Thangaraj et al. An Ab Initio Investigation on Relevant Oligomerization Reactions of Toluene Diisocyanate (TDI) Polymers 2022, 14, 4183.

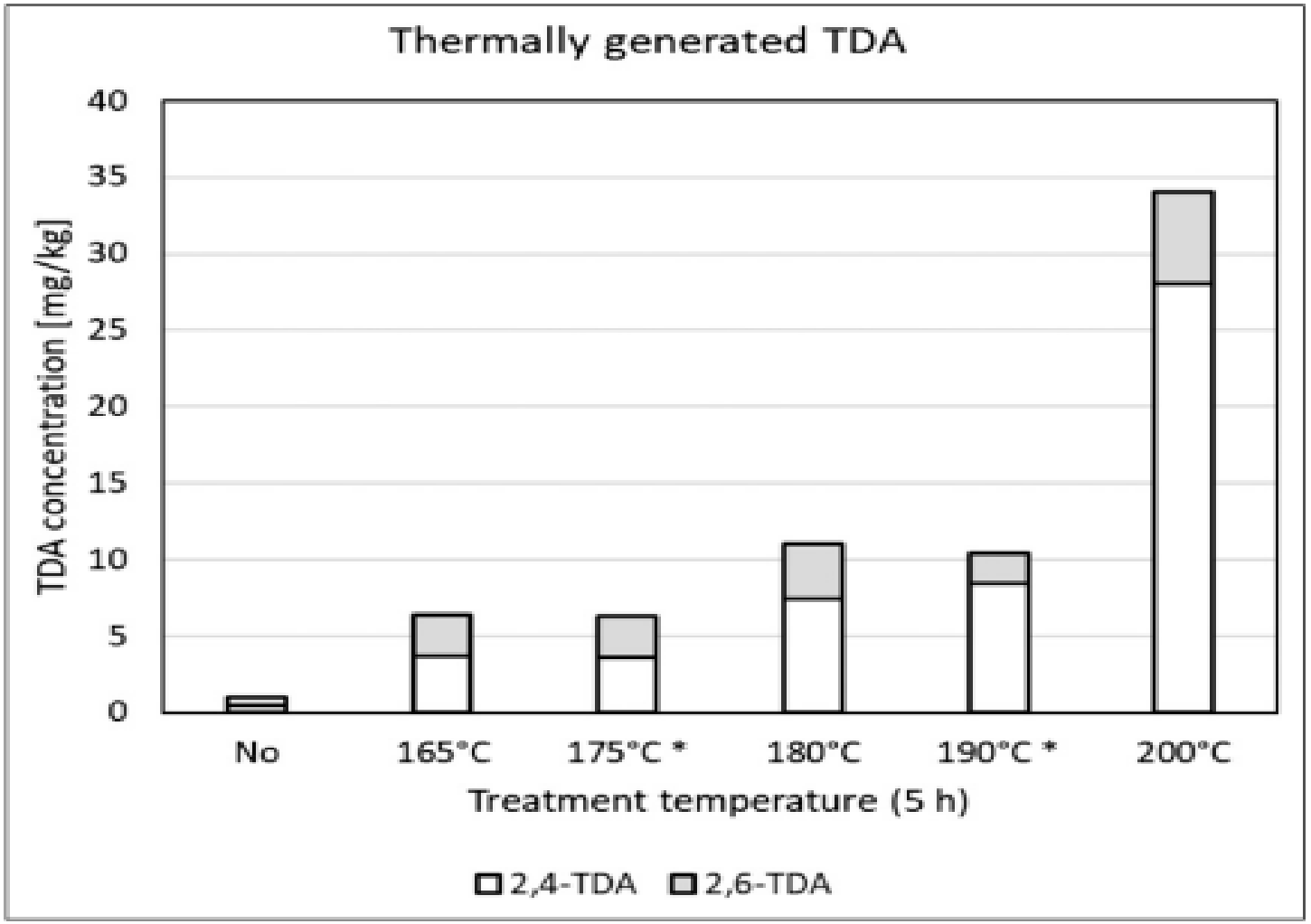


Approccio Green Haritz Sardon et al. *Synthesis of Polyurethanes Using Organocatalysis: A Perspective* DOI: 10.1021/acs.macromol.5b00384 Macromolecules 2015, 48, 3153−3165

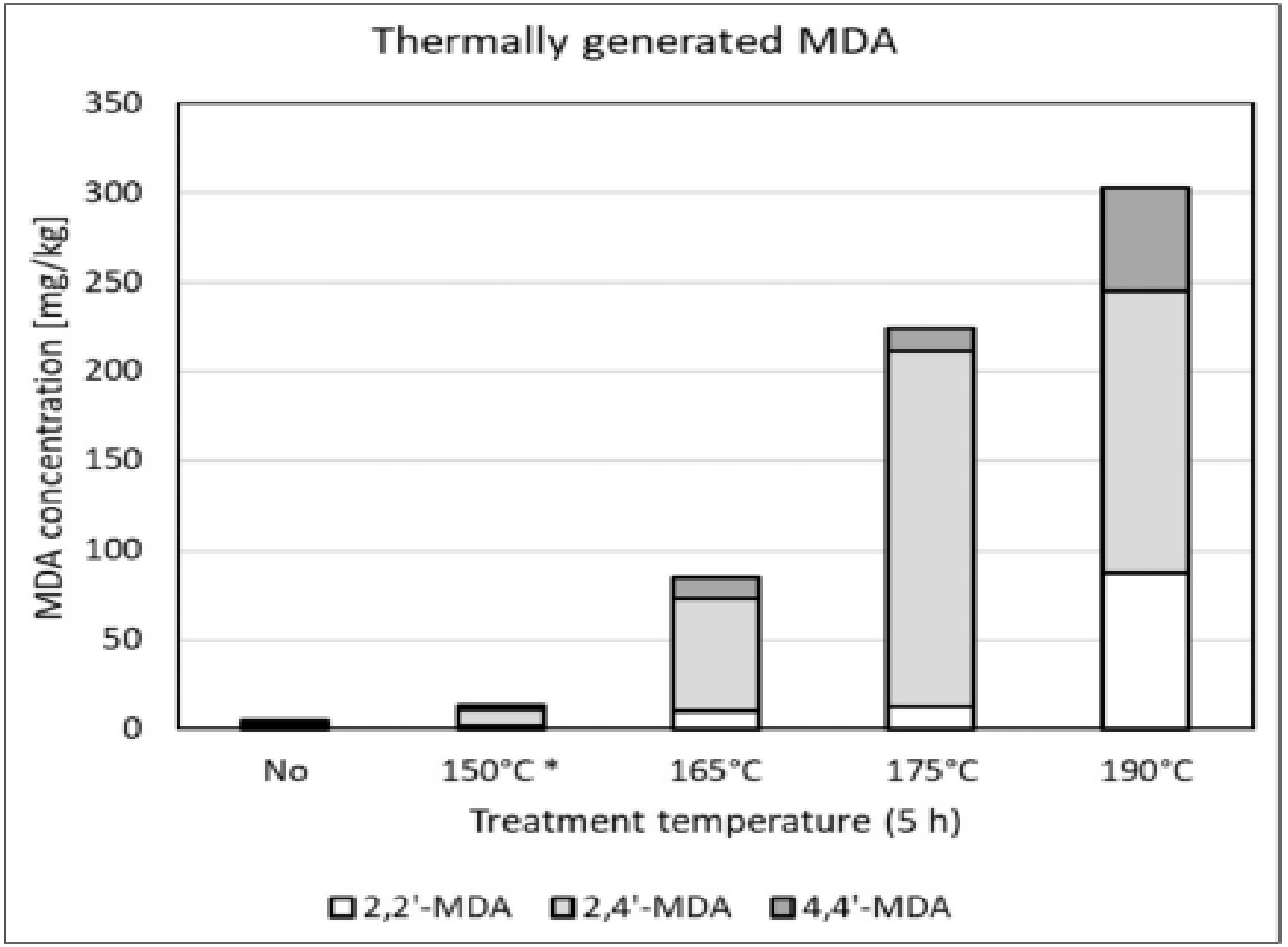
Approccio Green Haritz Sardon et al. *Synthesis of Polyurethanes Using Organocatalysis: A Perspective* DOI: 10.1021/acs.macromol.5b00384 Macromolecules 2015, 48, 3153−3165



Daniel Karlsson et al. *Emission and time-resolved migration rates of aromatic diamines from two flexible polyurethane foams* Toxicology and Industrial Health 2023, Vol. 39(6) 298–312

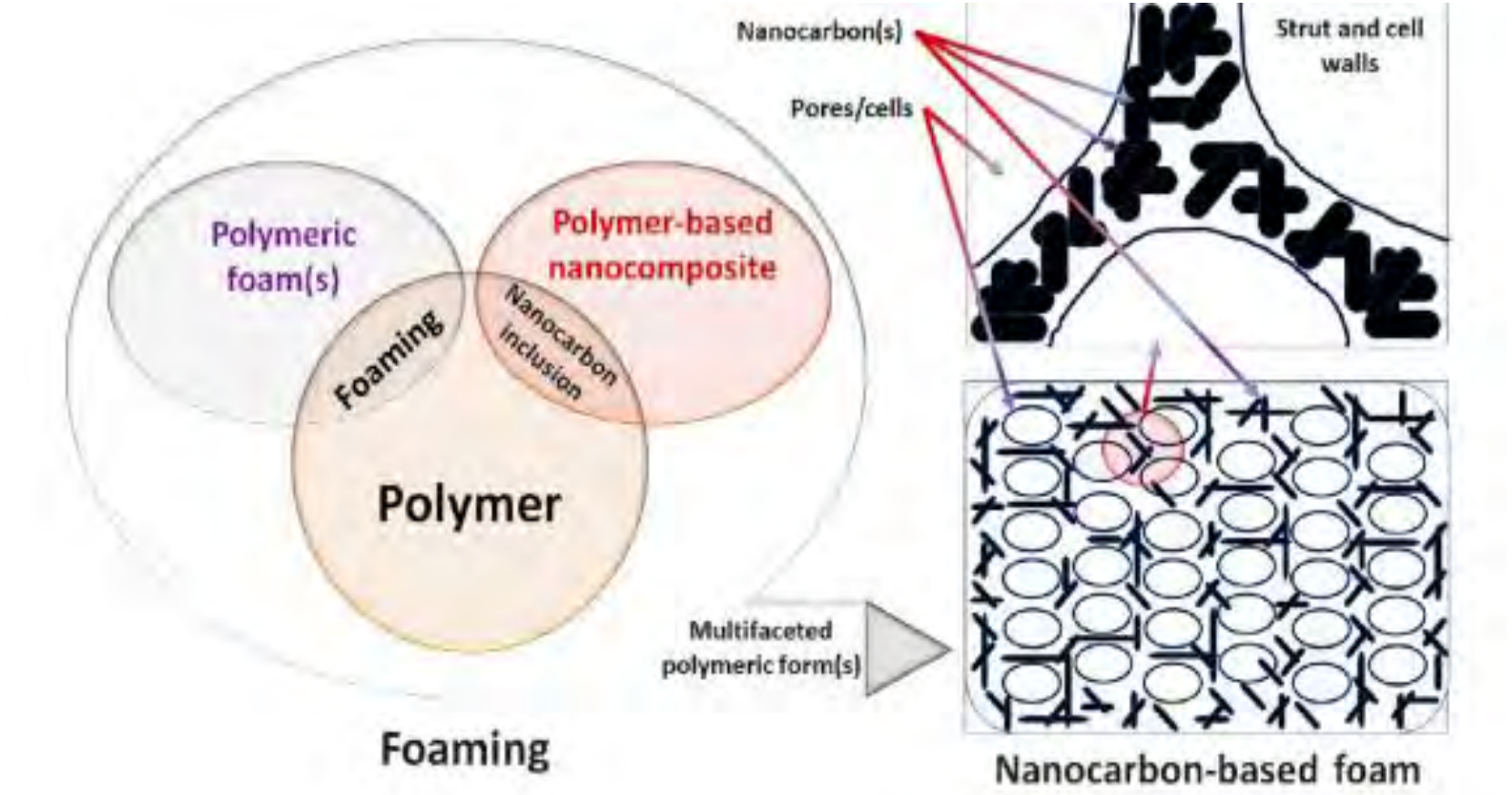


Daniel Karlsson et al. *Emission and time-resolved migration rates of aromatic diamines from two flexible polyurethane foams* Toxicology and Industrial Health 2023, Vol. 39(6) 298–312

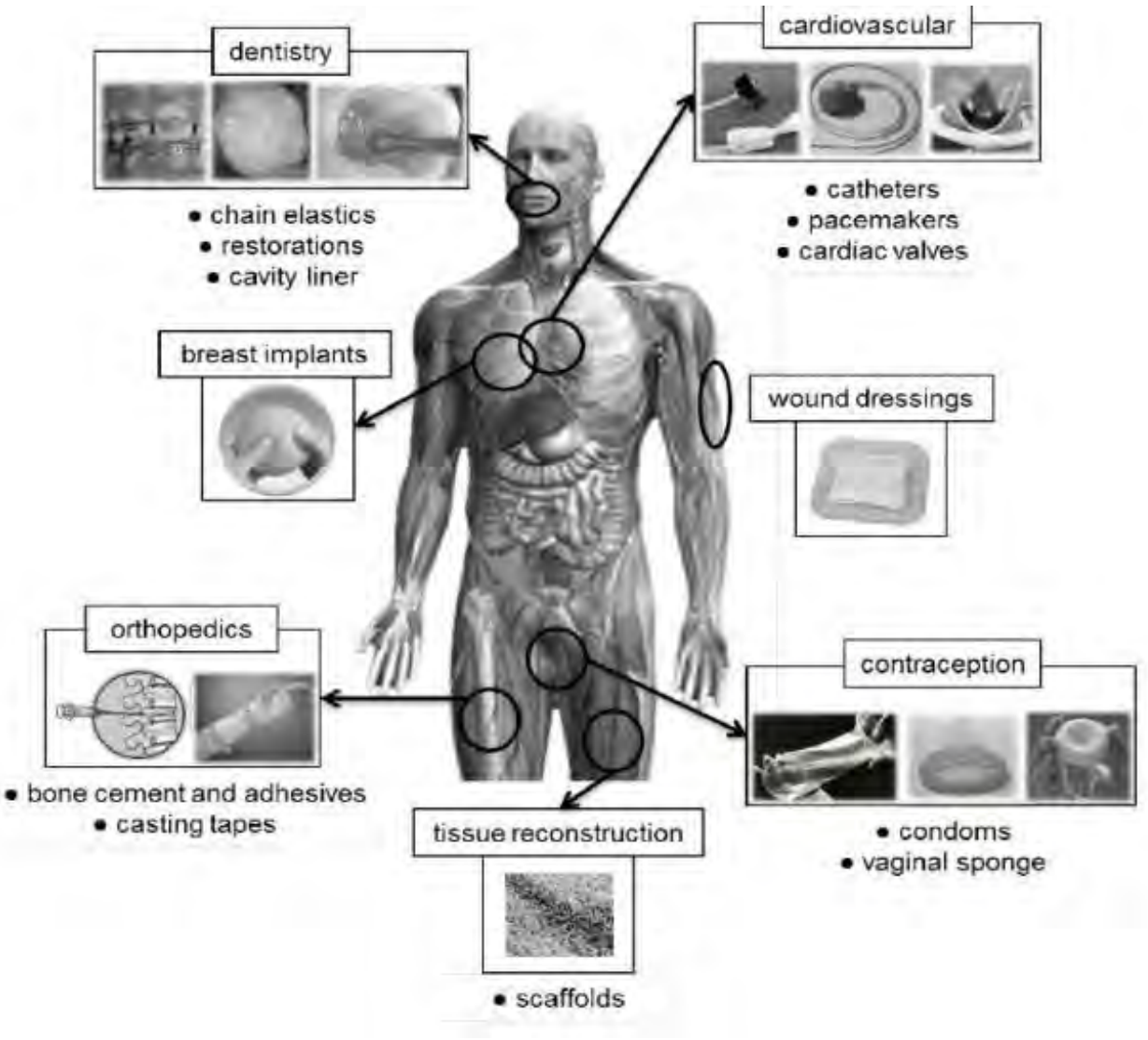


Nuove applicazioni

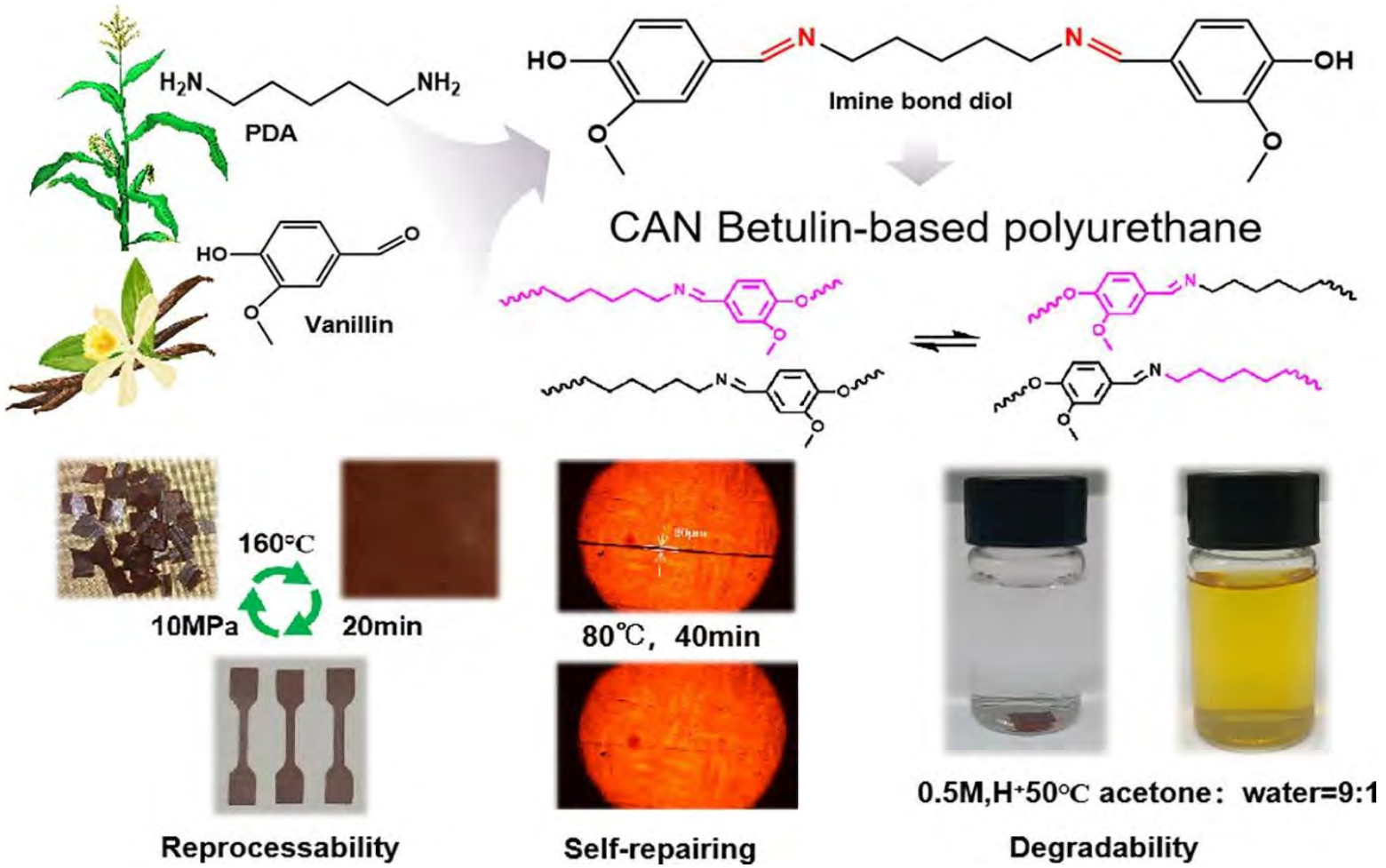
Ritima Banerjee et al. Nanocarbon-Containing Polymer Composite Foams: A Review of Systems for Applications in Electromagnetic Interference Shielding, Energy Storage, and Piezoresistive Sensors.*Industrial & Engineering Chemistry Research* **2023** *62* (18), 6807-6842
DOI: 10.1021/acs.iecr.3c00089



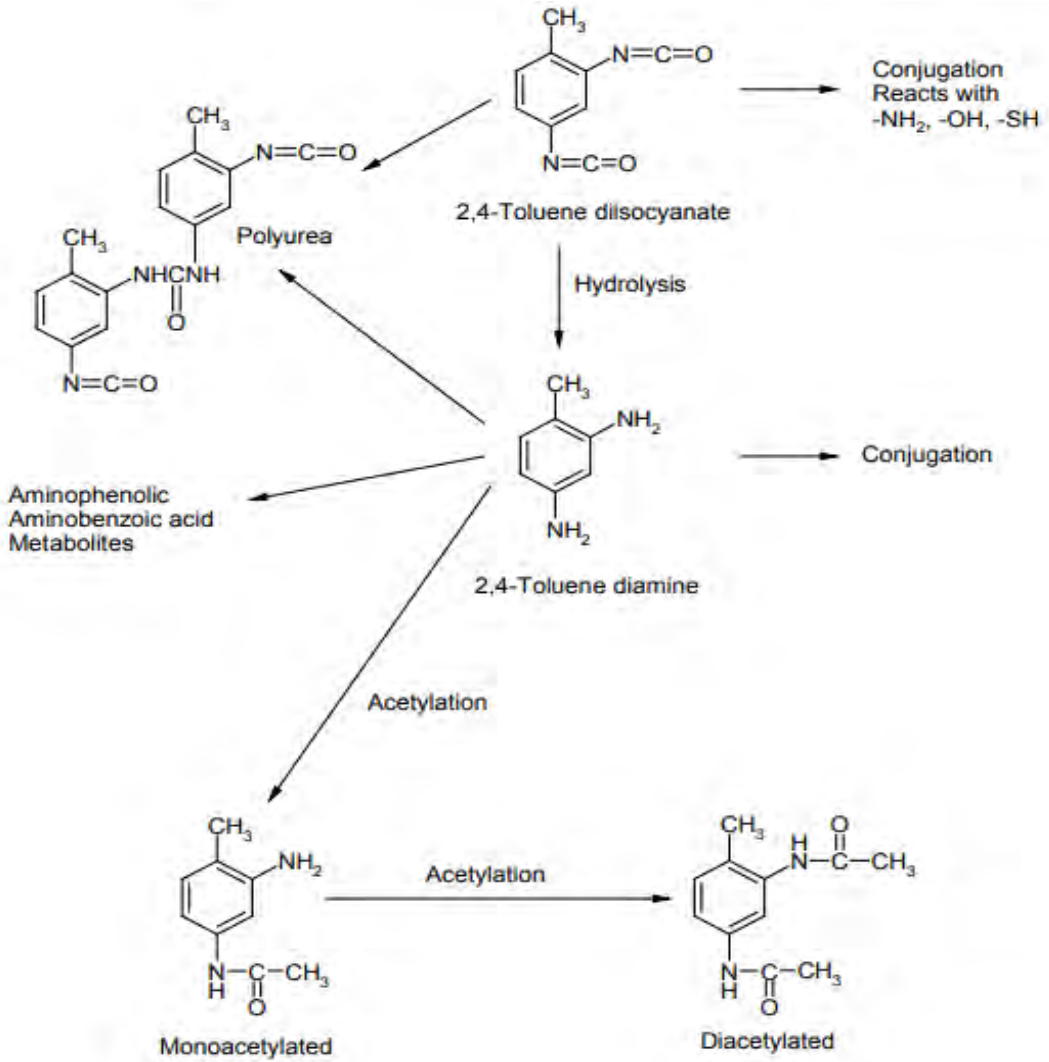
Fonte : tesi di laurea magistrale di Alice Girelli in Ingegneria Biomedica-Politecnico di Milano - Anno Accademico 2020-2021 “Espansi poliuretanici funzionalizzati con PIME per *wound-dressing*”



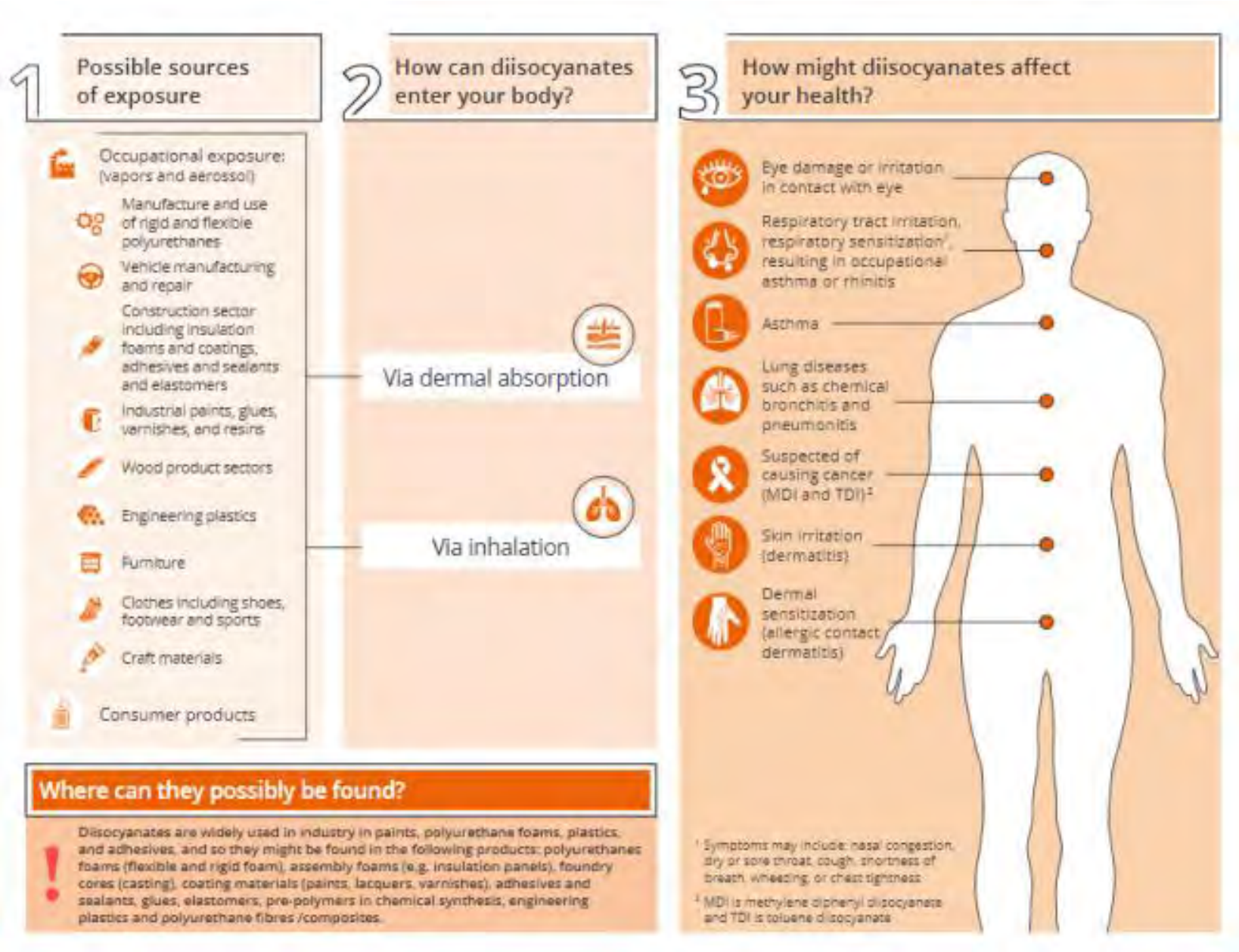
Xiaobo Xu et al. *Fully biomass-derived polyurethane based on dynamic imine with self-healing, rapid degradability, and editable shape memory capabilities*, Chemical Engineering Journal, Volume 479, 2024, 147823, ISSN 1385-8947,



Via metabolica *fonte:* Toxicological Profile For Toluene Diisocyanate And Methylenediphenyl Diisocyanate U.S. DEPARTMENT OF HEALTH AND HUMAN SERVICES .Public Health Service Agency for Toxic Substances and Disease Registry 2018



HBM4EU :Anilines and Diisocyanates. Substance report Tina Santonen et al. 2022



Opinion on scientific evaluation of occupational exposure limits for Diisocyanates

ECHA/RAC/A77-O-0000006826-64-01/F 2020

Derived Limit Values

<table>
<tr><td>OEL as 8-hour time weighted average (TWA) exposure:</td><td>A threshold for bronchial hyper-responsiveness or for the development of asthma, could not be observed.

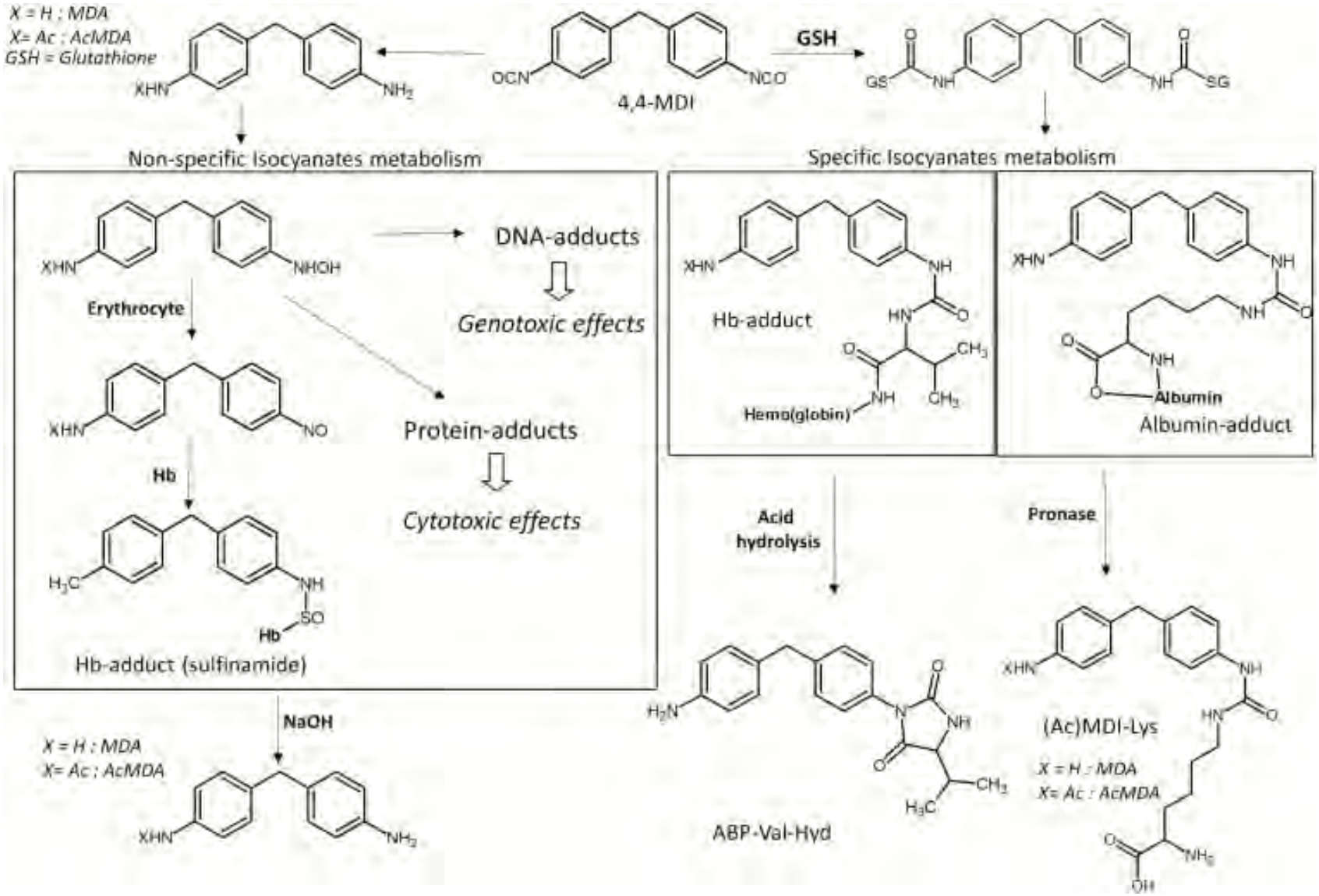
However, an OEL defined as an 8-hour time weighted average (TWA) exposure based on the 'NCO group' can be obtained from the exposure - excess risk relationships for hyperresponsiveness or diisocyanate asthma as derived below.

<table><tr><th>Excess risk over a working life period</th><th>Exposure - response relations derived from Pronk et al. (2009), and Collins et al. (2017), in µg/m³ NCO in air</th></tr><tr><td>0.1%</td><td><0.025</td></tr><tr><td>0.5%</td><td>0.027-0.040</td></tr><tr><td>1%</td><td>0.055-0.070</td></tr><tr><td>2%</td><td>0.12-0.19</td></tr><tr><td>3%</td><td>0.22-0.33</td></tr><tr><td>4%</td><td>0.40-0.48</td></tr><tr><td>5%</td><td>>0.67</td></tr></table></td></tr>
<tr><td>STEL:</td><td>A 15-minutes Short Term Exposure Limit (STEL) value which is maximally a factor 2 higher than a derived OEL based on the exposure - excess risk relation. This STEL value should not exceed 6 µg/m³ NCO.</td></tr>
<tr><td>BLV:</td><td>No BLV</td></tr>
<tr><td>BGV:</td><td>Set at the limits of quantification (LOQs) for relevant diisocyanate metabolites (diamines) in urine</td></tr>
</table>

Notations

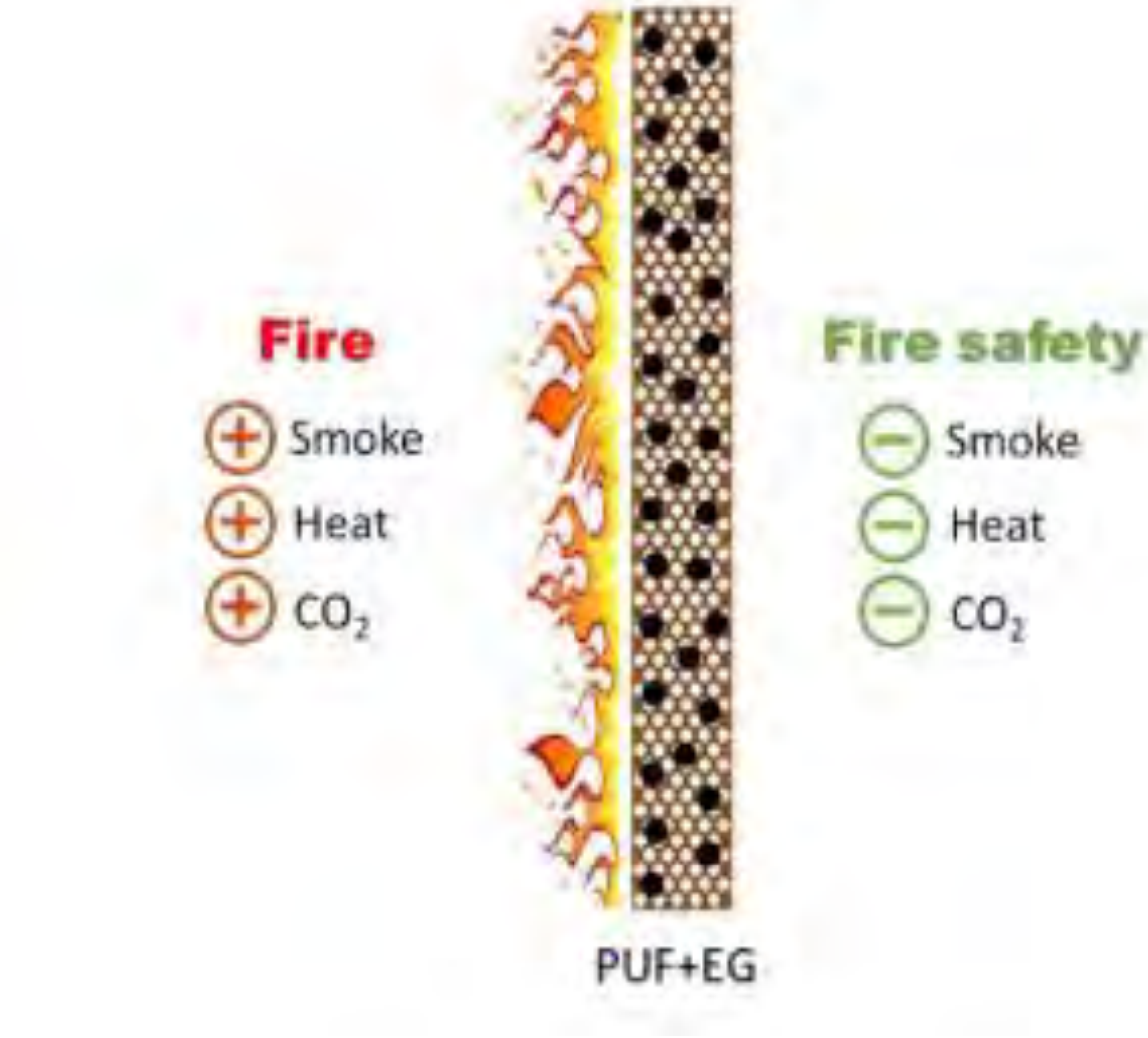
Notations:	skin sensitisation, respiratory sensitisation, 'skin'

Sholten B. et al. Biomonitoring for Occupational Exposure to Diisocyanates: A Systematic Review [Ann Work Expo Health.](#) 2020 Jul; 64(6): 569–585.



Effetti dei ritardanti di fiamma (expandable graphite EG)

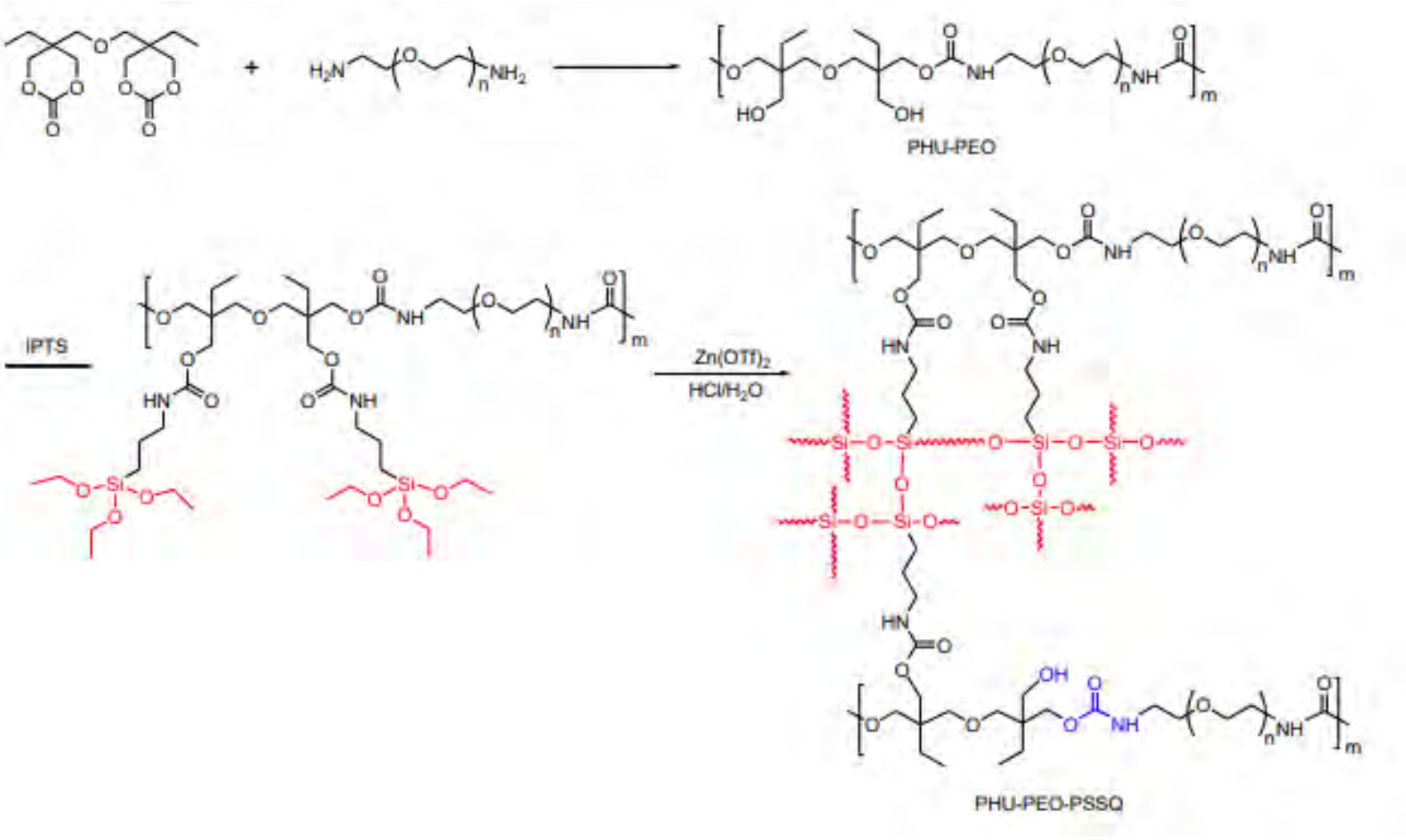
Nuno Vasco Costa, Gama Batista Melo Tesi: Functional bio-based polyurethane foams from industrial residues Universidade de Aveiro 2017 Departamento de Química



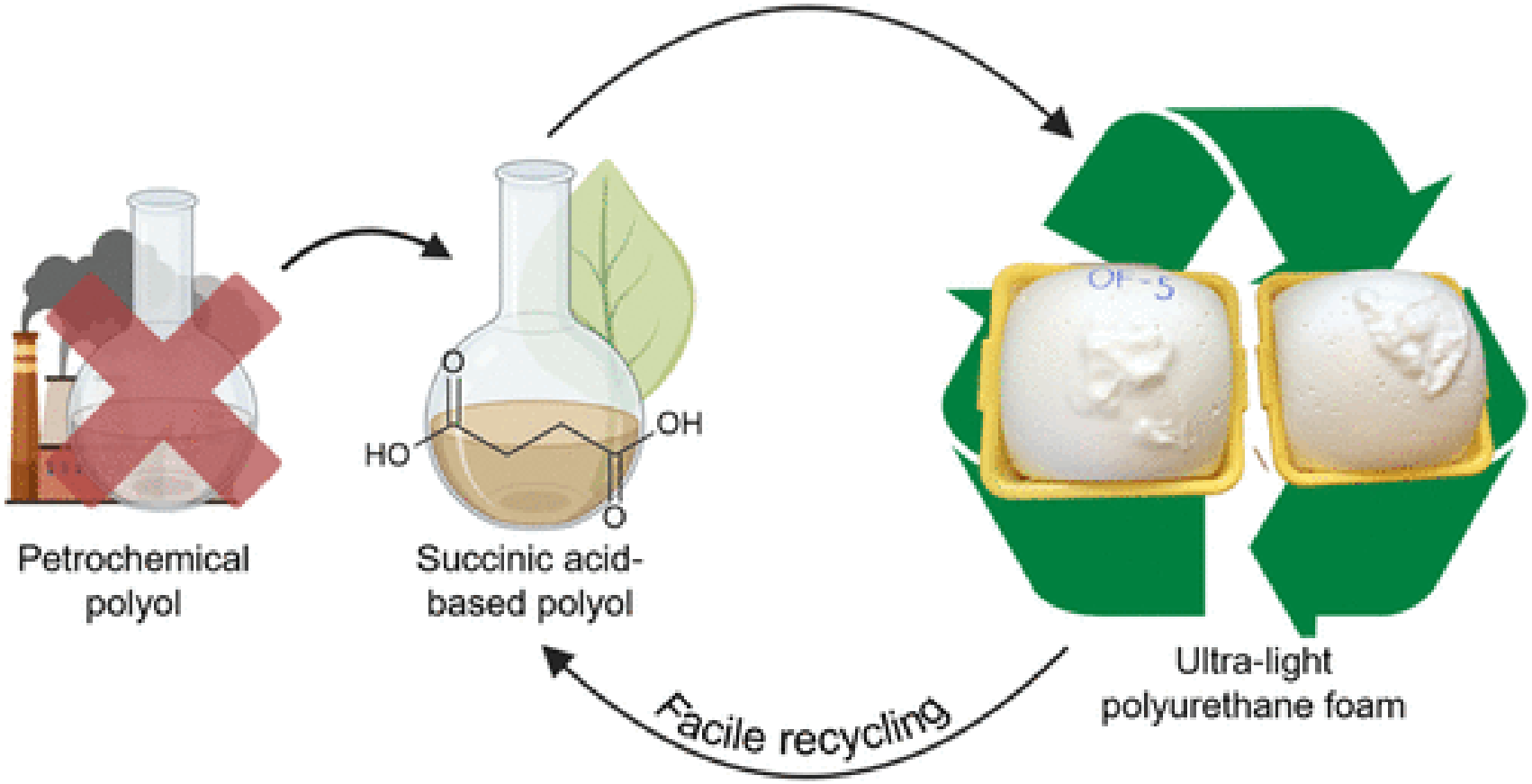
Kirill Cherednichenko et al. **Fireproof Nanocomposite Polyurethane Foams: A Review** Polymers 2023, 15, 2314. https://doi.org/10.3390/polym15102314

Halogen-Containing FRs	Phosphorous-Containing FRs	Nitrogen-Containing FRs	Other FRs
• Cycloaliphatic compounds (e.g., HET acid, TCPA, HCCPD, HBCDD, HBCD, TBPA) • Esters/ethers (PBDEs, DECA, DBDPO) • Derivatives of aromatic compounds (e.g., TBBPA, TBP, pentachlorophenol) • Other organic compounds (e.g., halogen-containing polyols and isocyanates, aliphatic compounds, polymers, paraffins)	• Elemental red phosphorous • Inorganic phosphates (e.g., APP) • Organic phosphates (including aryl phosphates (e.g., TPP), alkyl phosphates (e.g., TBP), bisphosphates (e.g., RDP, BDP)) • Chlorinated phosphates (e.g., TCEP, TCPP, TDCP) • Phosphinates (e.g., DEPAL) • Phosphine oxides (e.g., DOPO)	• Melamine and its derivatives (e.g., MCA, melamine homologs) • Inorganic compounds (e.g., APP, ammonium bibo-rate/pentaborate) • Organic compounds (e.g., urea, EUF)	• Metals hydroxides (e.g., ATH, MDH) • Metals salts (e.g., zinc borate, zinc stannate, zinc hydroxystannate, zinc carbonate, calcium carbonate, manganese carbonate, ammonium carbonate, antimony trioxide, arsenic oxide, calcium sulfate) • Expandable graphite • Carbon nanomaterials • Clays (e.g., montmorillonite, kaolinite)

Lei Li et al. *Polyhydroxyurethane and Poly(ethylene oxide) Multiblock Copolymer Networks: Crosslinking with Polysilsesquioxane, Reprocessing and Solid Polyelectrolyte Properties*
Polymers 2023, 15, 4634. https://doi.org/ 10.3390/polym1524463



Gotkiewicz, Olga et al. **Biobased Ultralow-Density Polyurethane Foams with Enhanced Recyclability** *ACS Sustainable Chem. Eng.* 2024, 12, 4, 1605-1615



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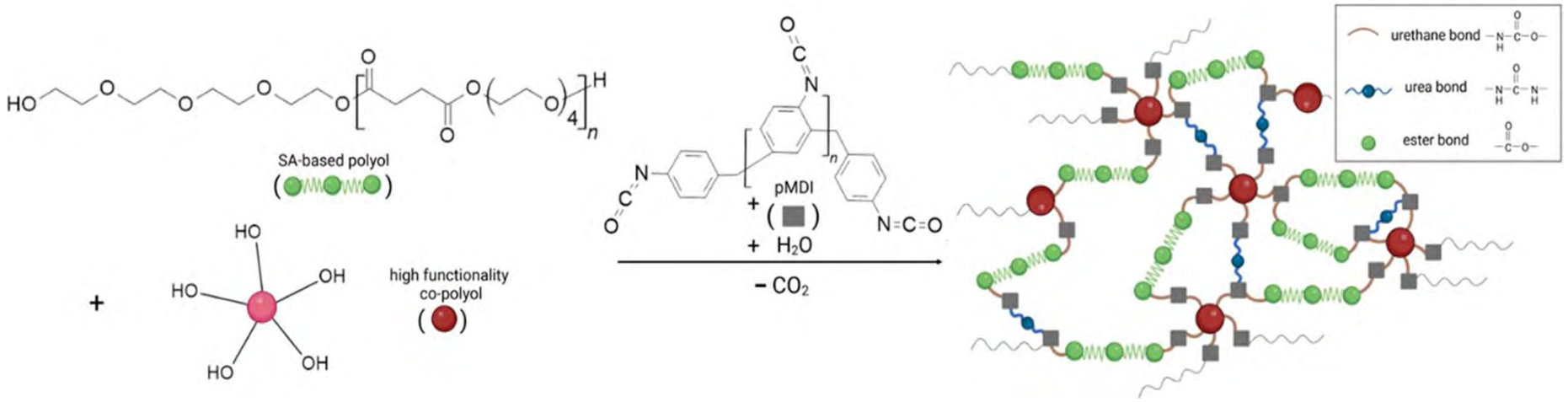
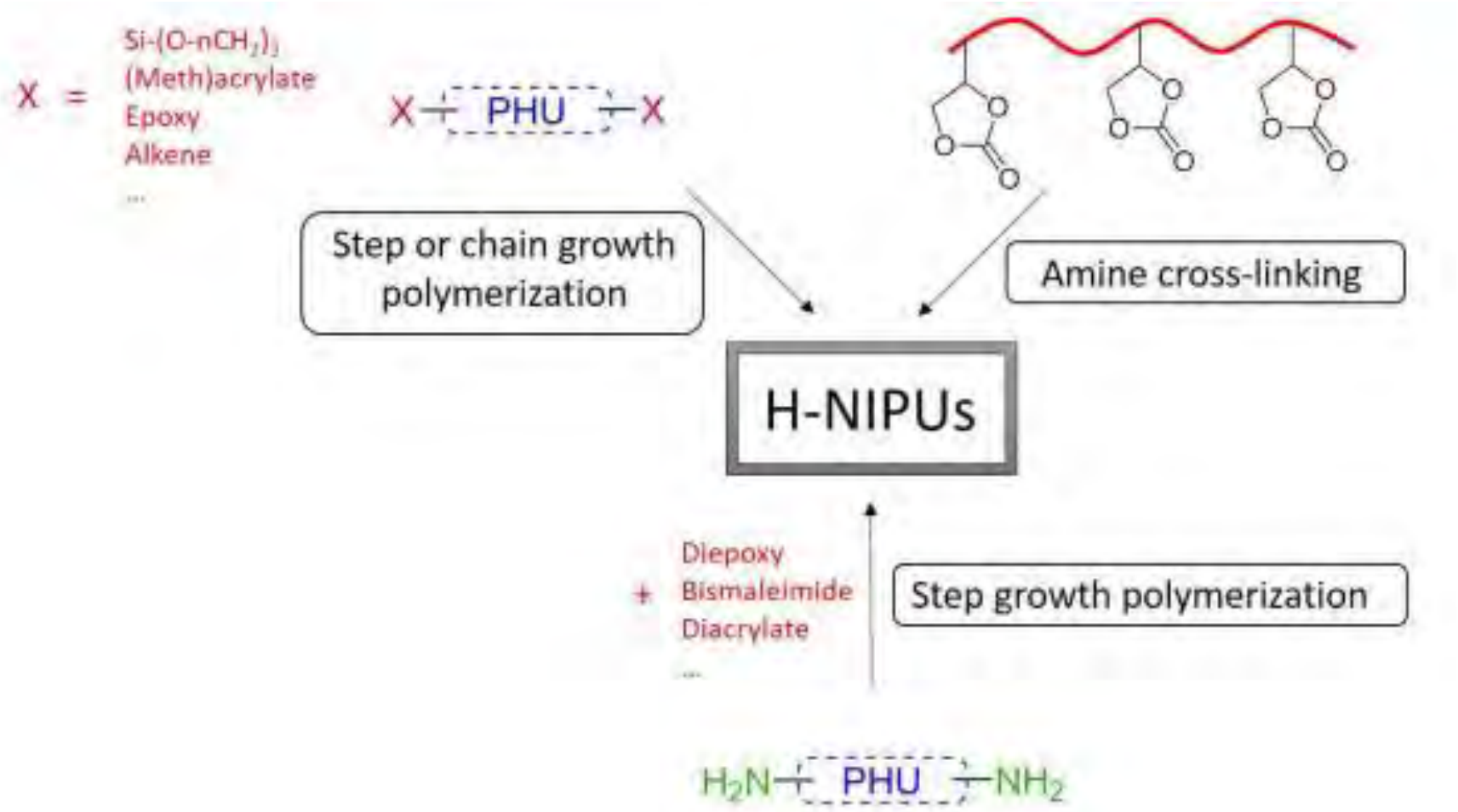
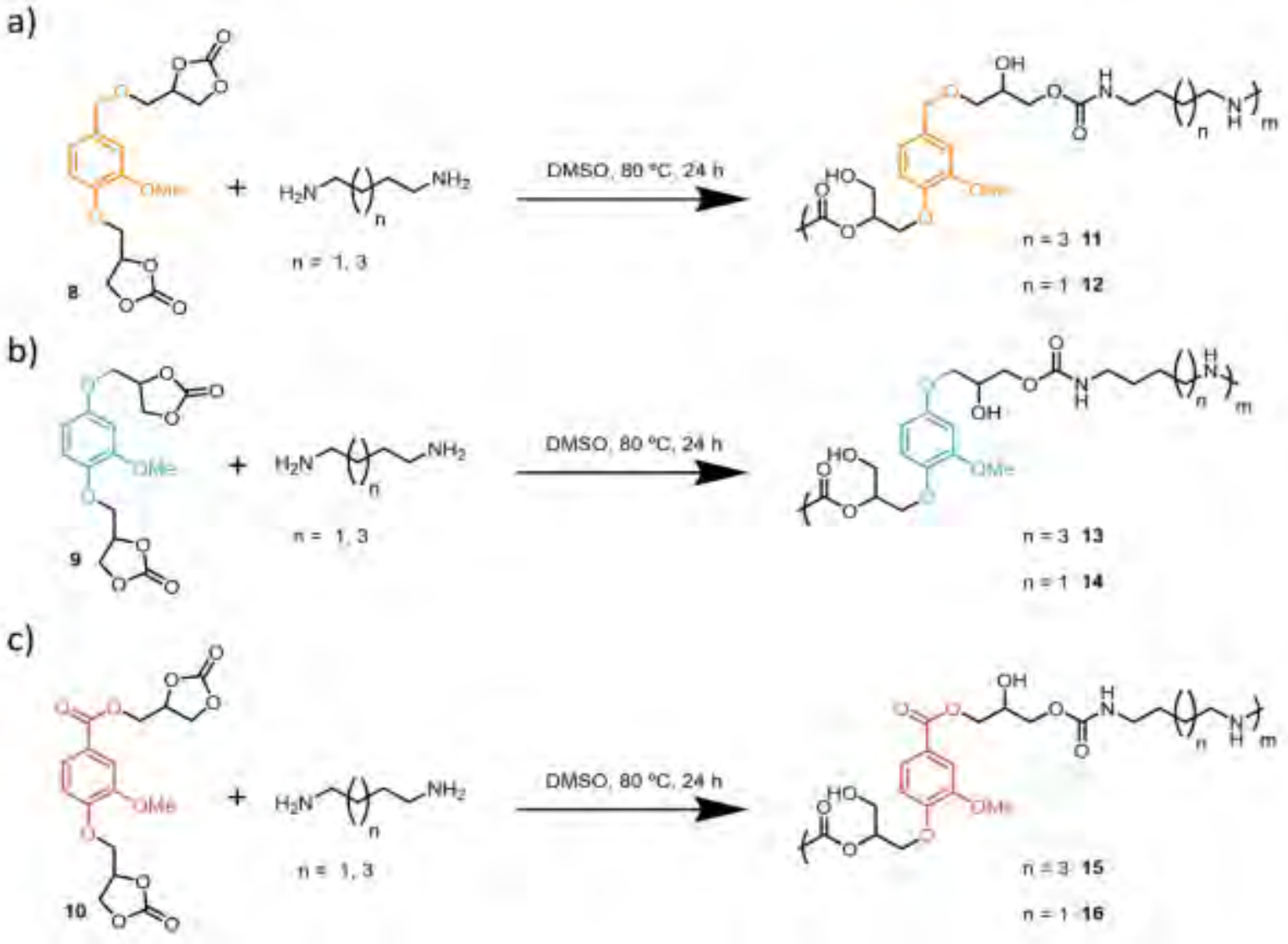


Yvan Ecochard, Sylvain Caillol *Hybrid Polyhydroxyurethanes: how to overcome limitations and reach cutting edge properties?* ICGM, Univ Montpellier, CNRS, ENSCM, Montpellier, France Manuscript_e63de756adcc005b843feb221d14c534 2020

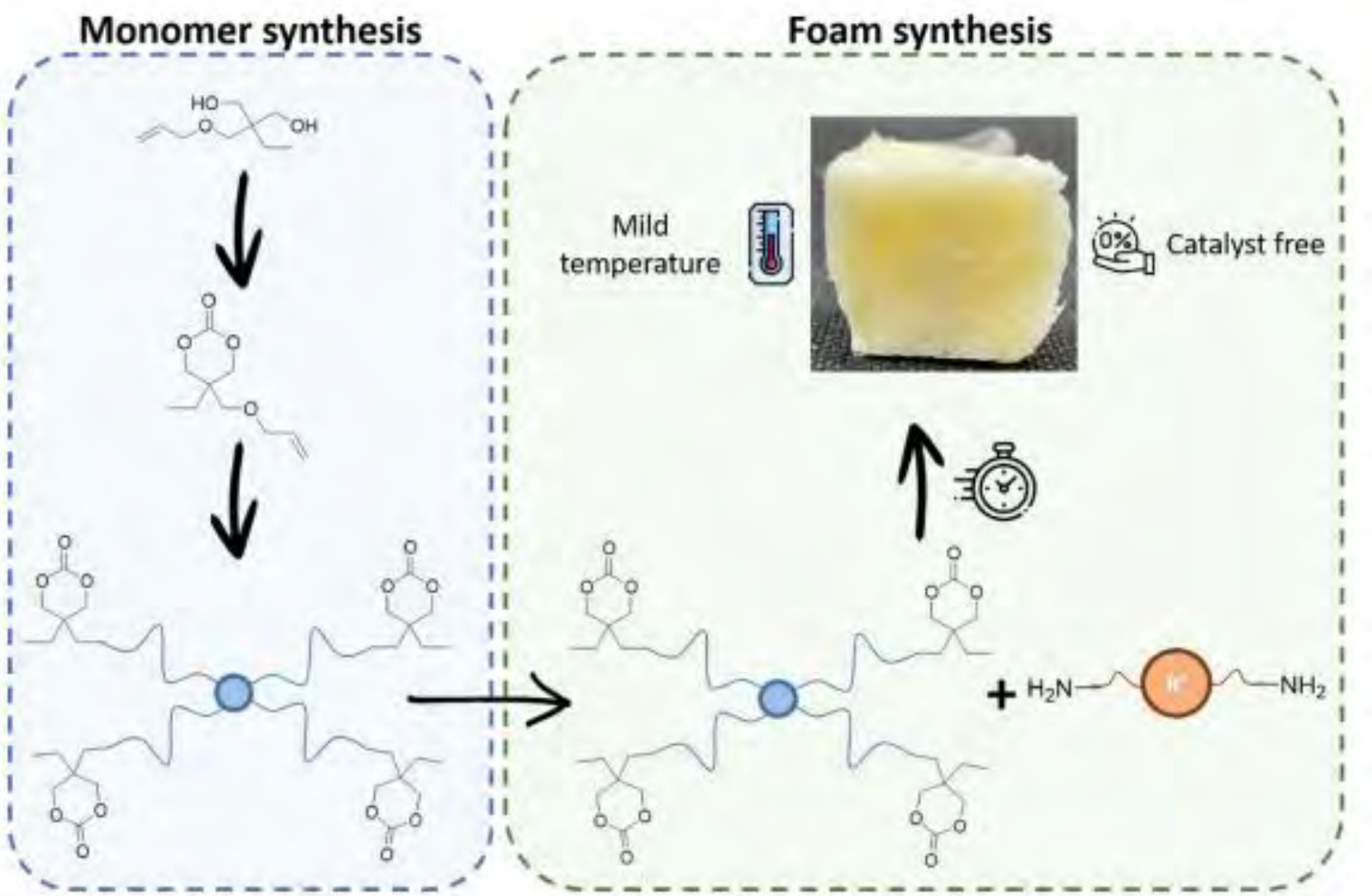


Noé Fanjul-Mosteirín et al. **Bio-based non-isocyanate poly(hydroxy urethane)s (PHU) derived from vanillin and CO2**

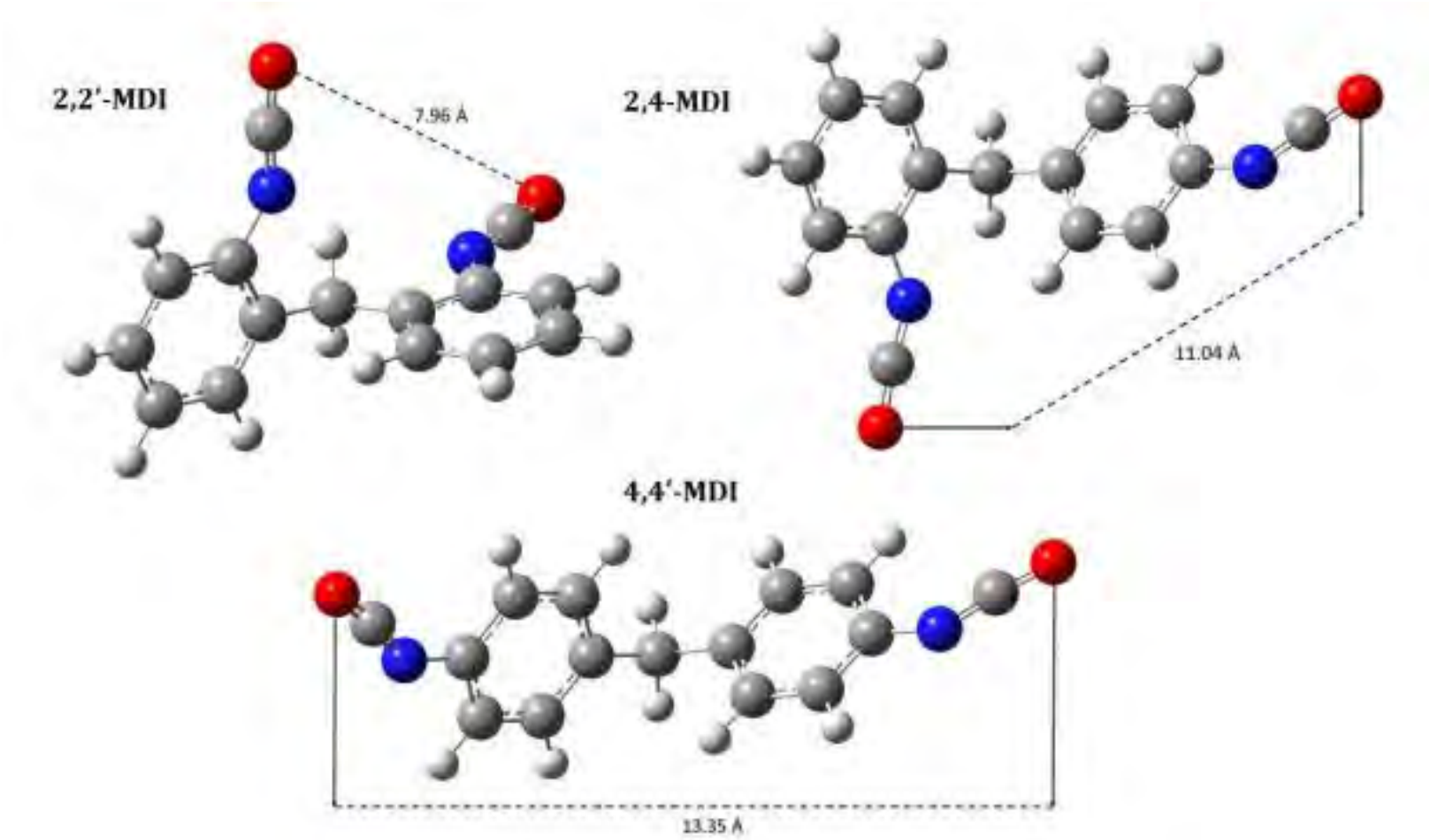
Mater. Adv., 2023, 4, 2437–2448 | 2437



Guilhem Coste et al. *Non-isocyanate Polyurethane Foams based on Six-membered cyclic Carbonates* European Polymer Journal, 2022, 176, pp.111392. ff10.1016/j.eurpolymj.2022.111392ff. ffhal-03719091



Renáta Zsanett Boros *A Study of Elementary Reactions of Isocyanate Production*
Ph.D. Dissertation Institute of Chemistry University of Miskolc ,Hungary 2019



Market volume of polyurethane worldwide from 2015 to 2022, with a forecast for 2023 to 2030 (in million metric tons)