FRIEDRICH-ALEXANDER UNIVERSITÄT ERLANGEN-NÜRNBERG

crt Lehrstuhl für Chemische Reaktionstechnik Professur für Katalytische Materialien

Synthese und Modifikation von Kohlenstoffen für katalytische Anwendungen

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FRIEDRICH-ALEXANDER UNIVERSITÄT ERLANGEN-NÜRNBERG

ENGINEERING OF ADVANCED MATERIALS

crt Lehrstuhl für Chemische Reaktionstechnik Professur für Katalytische Materialien

Outline

Why carbon in catalysis?

Why tuning carbon catalyst properties?

Possibilities for precise control of carbon catalyst properties

Two unconventional examples and success stories

Versatile material

6

C

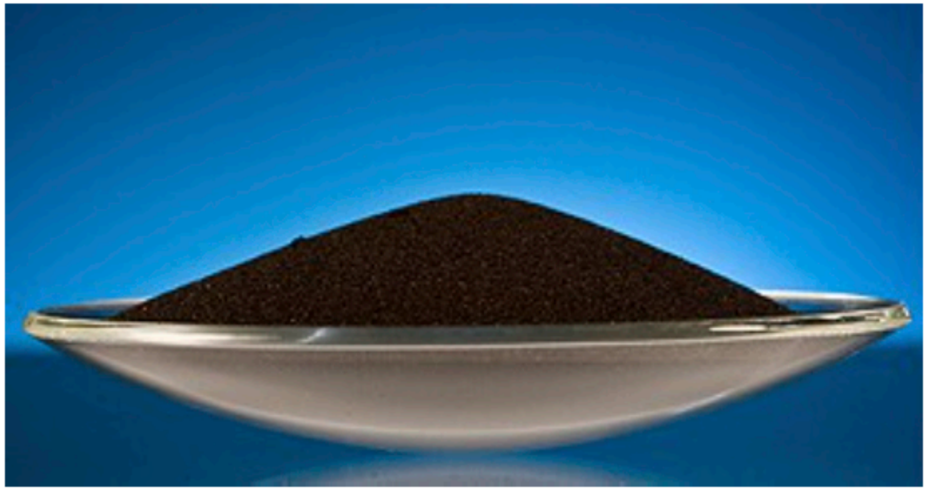
Carbon

12.0107

Carbon in electrocatalysis

Spezialkatalysatoren für Brennstoffzellen

Der Wasserstoff- und Brennstoffzellentechnologie kommt in der Zukunft aus umwelt- und energiepolitischer Sicht wachsende Bedeutung zu. Neben den Anwendungen im mobilen Bereich finden Brennstoffzellensysteme auch bei stationären Systemen, wie etwa bei der Hausenergieversorgung zunehmendes Interesse. Im portablen Bereich gibt es bereits marktreife Systeme für die unterschiedlichsten Einsatzfelder.



Moderne Brennstoffzellentechnologie, wie etwa PEMFC, benötigen hocheffiziente Elektrokatalysatoren für die Membranbeschichtung.

Typ	[BET-Oberfläche m²/g]	Partikelgröße D 50 [µm]
40 % Pt/Ruß	150	5
20 % Pt/Ruß	150	5
40 % Pt-Ru/Ruß	150	5
10 % Pt/Ruß	800	10
20 % Pt/Ruß	800	10

http://heraeus-katalysatoren.de

Carbon in thermal catalysis

Johnson Matthey
Catalysts

Heterogeneous Catalysts Application Table

	20% Pd/C	10% Pd/C					5% Pd/C					
	1	2	3	4	5	6	7	8	9	10	11	12
	20R91	10R87L	10R374	10R39	10R394	10R487	5R58	5R87L	5R434	5R393	5R394	5R338M
Hydrogenation of Acetylenes to Olefins												
Hydrogenation of Vinyl Acetylenes to Dienes												
Hydrogenation of Acetylenes to Alkanes	•	•	•	•	•	•	•	•	•	•	•	•
Hydrogenation of Olefins to Alkanes	•	•	•	•	•	•	•	•	•	•	•	•
Hydrogenation of Diolefins to Monolefins												
Hydrogenation of Aromatics to Cycloalkanes	•	•		•	•	•						
Hydrogenation of Aromatics to Partially Hydrogenated Aromatics	•	•				•	•	•	•			•
Hydrogenation of Phenols to Cyclohexanones	•	•	•	•	•	•	•	•	•	•	•	•
Hydrogenation of Phenols to Cyclohexanols												
Hydrogenation of Benzoic acids to Cyclohexane Carboxylic Acids		•				•		•	•			•
Hydrogenation of Anilines to Cyclohexyl Amines												
Hydrogenation of Pyridines to Piperidines	•	•		•	•	•	•	•	•	•	•	•
Hydrogenation of Quinolines	•	•		•	•	•	•	•	•	•	•	•
Hydrogenation of Pyrroles to Pyrrolidines												
Hydrogenation of Indoles												
Hydrogenation of Furans to Tetrahydrofurans	•	•	•	•	•	•	•	•	•	•	•	•

Carbon in thermal catalysis

Johnson Matthey Catalysts

Heterogeneous Catalysts Application Table

	20% Pd/C	10% Pd/C					5% Pd/C								3% Pd/C	5% Pd/Al_2O_3	5% Pd/$CaCO_3$	5% Pd/$BaSO_4$	2% Pd/SIO_2-Al_2O_3	5% Pd, Pb/$CaCO_3$	2.5% Pd, 2.5% Pt/C	10% Pt/C	5% Pt/C				5% Pt/graphite	~1% Pt/C		5% Pt(B)/C	5% Pt(S)/C	5% Pt/Al_2O_3	5% Rh/C		5% Rh/Al_2O_3	5% Ru/C		5% Ru/Al_2O_3	5% Ru, 0.25% Pd/C	5% Ir/$CaCO_3$
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40
	20R91	10R87L	10R374	10R39	10R394	10R487	5R58	5R87L	5R434	5R393	5R394	5R338M	5R39	5R37	3R38H	5R325	5R405	5R29A	2R31	A305060-5	5R122	10R128M	5R18	5R103	5R117	5R128M	5R289	1R18MA	1.5R199	5R160	B106032-5	5R94	5R20A	5R592	5R524	5R97	5R619	5R698	5R611	5R30
Hydrogenation of Acetylenes to Olefins																	•	•		•																				
Hydrogenation of Vinyl Acetylenes to Dienes																	•	•		•																				
Hydrogenation of Acetylenes to Alkanes	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•																								
Hydrogenation of Olefins to Alkanes	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•																								
Hydrogenation of Diolefins to Monolefins																•				•																				
Hydrogenation of Aromatics to Cycloalkanes	•	•		•	•	•																•	•	•	•	•						•	•	•	•	•	•	•	•	
Hydrogenation of Aromatics to Partially Hydrogenated Aromatics	•	•				•	•	•	•			•			•	•						•	•	•	•	•										•	•	•	•	
Hydrogenation of Phenols to Cyclohexanones	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•																	•	•	•					
Hydrogenation of Phenols to Cyclohexanols																																	•	•	•	•	•	•	•	
Hydrogenation of Benzoic acids to Cyclohexane Carboxylic Acids		•				•		•	•			•			•							•	•	•	•	•							•	•	•	•	•	•	•	
Hydrogenation of Anilines to Cyclohexyl Amines																																	•	•	•	•	•	•	•	
Hydrogenation of Pyridines to Piperidines	•	•		•	•	•	•	•	•	•	•	•	•	•	•	•						•	•	•	•	•							•	•	•	•	•	•		
Hydrogenation of Quinolines	•	•		•	•	•	•	•	•	•	•	•	•	•	•							•	•	•	•	•							•	•	•	•	•	•		
Hydrogenation of Pyrroles to Pyrrolidines																						•	•	•	•	•							•	•	•					
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Hydrogenation of Aromatic Aldehydes to Alcohols	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•																								
Hydrogenation of Haloaromatic Aldehydes to Haloaromatic Alcohols																							•	•	•	•	•	•	•		•	•								
Hydrogenation of Aromatic Aldehydes to Hydrocarbons	•	•		•	•	•	•	•	•	•	•	•	•	•	•	•																								
Hydrogenation of Aliphatic Aldehydes to Alcohols																						•	•	•	•	•	•	•	•			•	•	•	•	•	•	•	•	•
Hydrogenation of α-β-Unsaturated Aldehydes to Saturated Alcohols																						•	•	•	•	•		•	•			•								
Hydrogenation of α-β-Unsaturated Aldehydes to Unsaturated Alcohols																											•													•
Hydrogenation of Aromatic Ketones to Alcohols	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•																								
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Hydrogenation of Lactones																						•	•	•	•	•		•	•			•				•	•	•	•	
Hydrogenation of Esters																																				•	•	•	•	
Hydrogenation of Carboxylic Acids																																				•	•	•	•	
Hydrogenation of Nitroaromatic Compounds to Amines	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•			•								
Hydrogenation of Halonitroaromatic Compounds to Aromatic Haloamines																							•	•	•	•	•	•	•		•									•
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Hydrogenation of Nitroaromatic Compounds to Aminophenols																											•	•	•		•									
Hydrogenation of Nitroaromatic Compounds to Azo Compounds	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•					•		•	•	•	•	•	•	•											
Hydrogenation of Nitroaromatic Compounds to Benzidines	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•					•		•	•	•	•	•	•	•											
Hydrogenation of Halonitroaromatic Compounds to Halobenzidines																							•	•	•	•	•	•	•											
Hydrogenation of Nitrosoaromatic Compounds to Amines		•	•			•		•	•	•					•	•					•	•	•	•	•	•	•	•	•											
Hydrogenation of Aliphatic Nitro Compounds to Amines	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•					•	•	•	•	•	•	•	•	•											
Hydrogenation of Aliphatic Nitroso Compounds to Amines		•	•			•		•	•			•			•	•					•	•	•	•	•	•	•	•	•											

Carbon in thermal catalysis

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Johnson Matthey Catalysts

Heterogeneous Catalysts Application Table

	20% Pd/C	10% Pd/C					5% Pd/C								3% Pd/C	5% Pd/Al_2O_3	5% Pd/$CaCO_3$	5% Pd/$BaSO_4$	2% Pd/SiO_2-Al_2O_3	5% Pd, Pb/$CaCO_3$	2.5% Pd, 2.5% Pt/C	10% Pt/C	5% Pt/C				5% Pt/graphite	~1% Pt/C		5% Pt(B)/C	5% Pt(S)/C	5% Pt/Al_2O_3	5% Rh/C		5% Rh/Al_2O_3	5% Ru/C		5% Ru/Al_2O_3	5% Ru, 0.25% Pd/C	5% Ir/$CaCO_3$
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40
	20R91	10R87L	10R374	10R39	10R394	10R487	5R58	5R87L	5R434	5R393	5R394	5R338M	5R39	5R37	3R38H	5R325	5R405	5R29A	2R31	A305060-5	5R122	10R128M	5R18	5R103	5R117	5R128M	5R289	1R18MA	1.5R199	5R160	B106032-5	5R94	5R20A	5R592	5R524	5R97	5R619	5R698	5R611	5R30
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Hydrogenation of Lactones																						•	•	•	•	•		•	•			•				•	•	•	•	
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Hydrogenation of Halonitroaromatic Compounds to Halobenzidines																							•	•	•	•	•	•	•											
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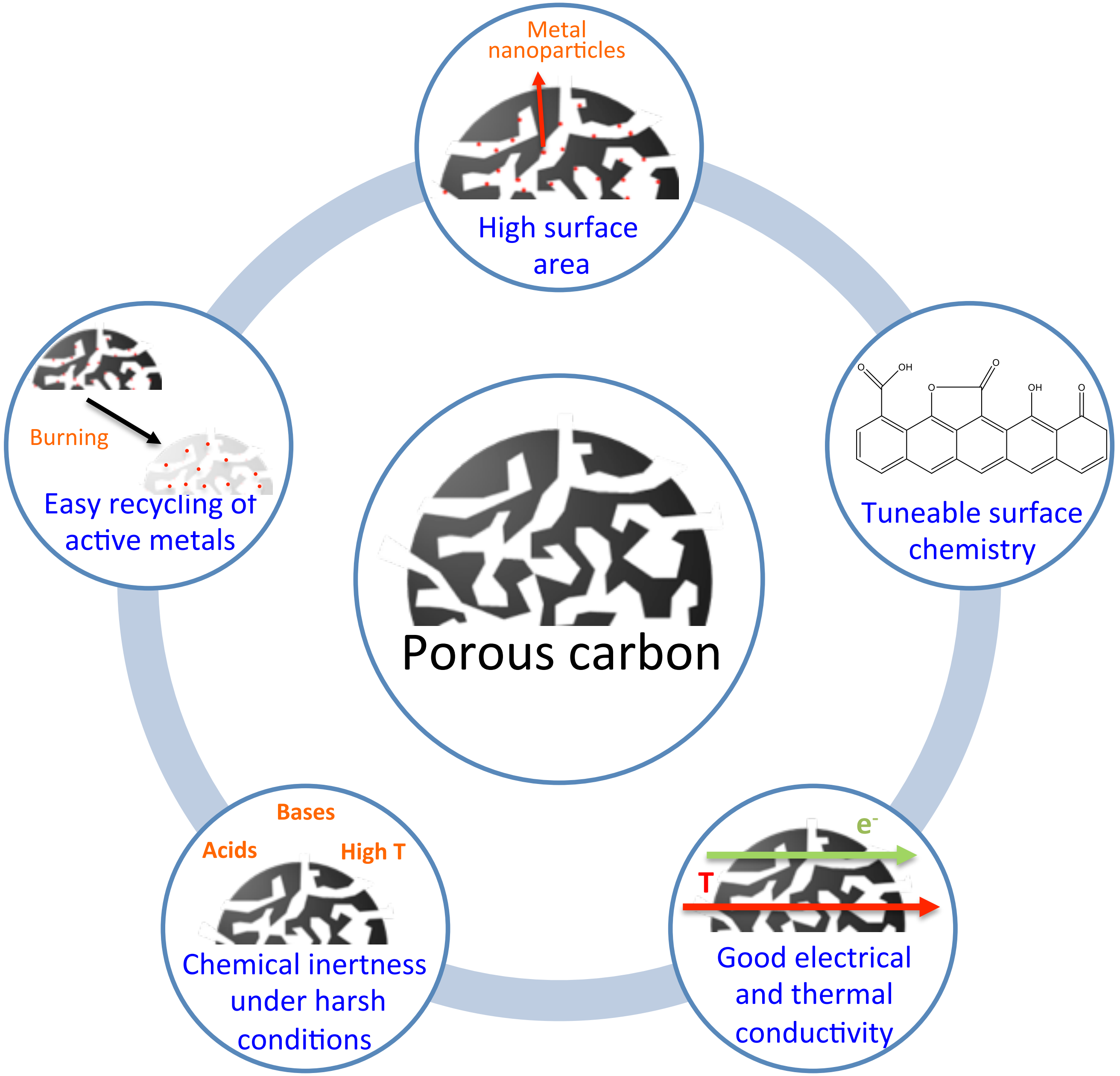
Hydrogenation of Aliphatic Nitro Compounds to Amines	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•					•	•	•	•	•	•	•	•	•											
Hydrogenation of Aliphatic Nitroso Compounds to Amines		•	•			•		•	•			•			•	•					•	•	•	•	•	•	•	•	•											

Why carbon in catalysis?

Trägermaterialien und deren Eigenschaften:			
Träger	**BET [m^2/g]**	**Schüttdichte [g/l]**	**Porenvolumen (ml/g)**
Aktivkohle	800-1500	250-500	0,5 - 2,5
Al_2O_3	1-400	450-1800	0,1 - 1,0
Al_2O_3-SiO_2	150-350	400-800	0,1 - 0,6
SiO_2	150-250	400-600	0,5 - 1,5
TiO_2	50-100	500-1100	0,2 - 0,6
Ce/ZrO_2	50-200	900-1200	---
Zeolithe	200-650	400-600	0,5 - 0,8

http://heraeus-katalysatoren.de

Why carbon in catalysis?

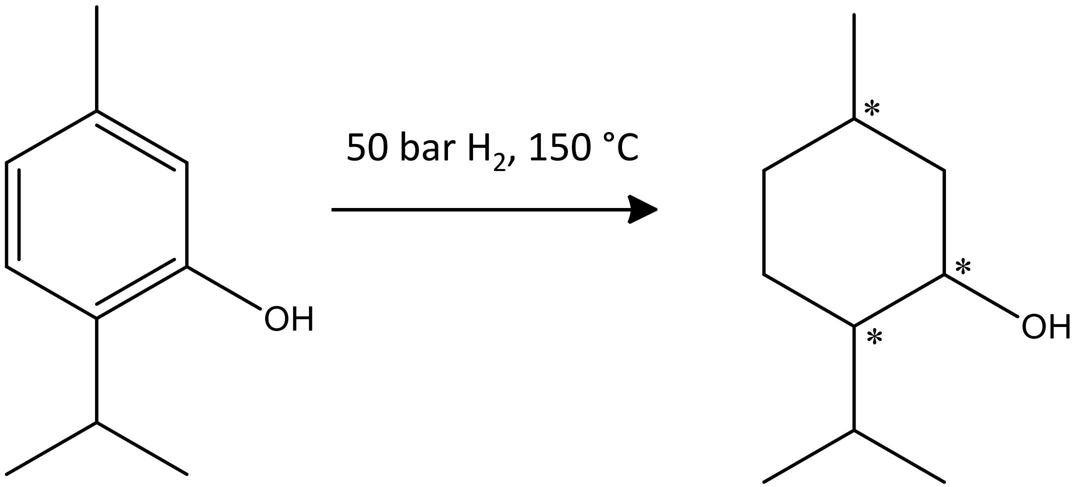


Why tuning carbon catalysts properties?

Let's see results from two instructive examples.

Application of carbonaceous catalysts: Menthol

- Carbon supported catalysts are used for the hydrogenation of thymol for menthol production.



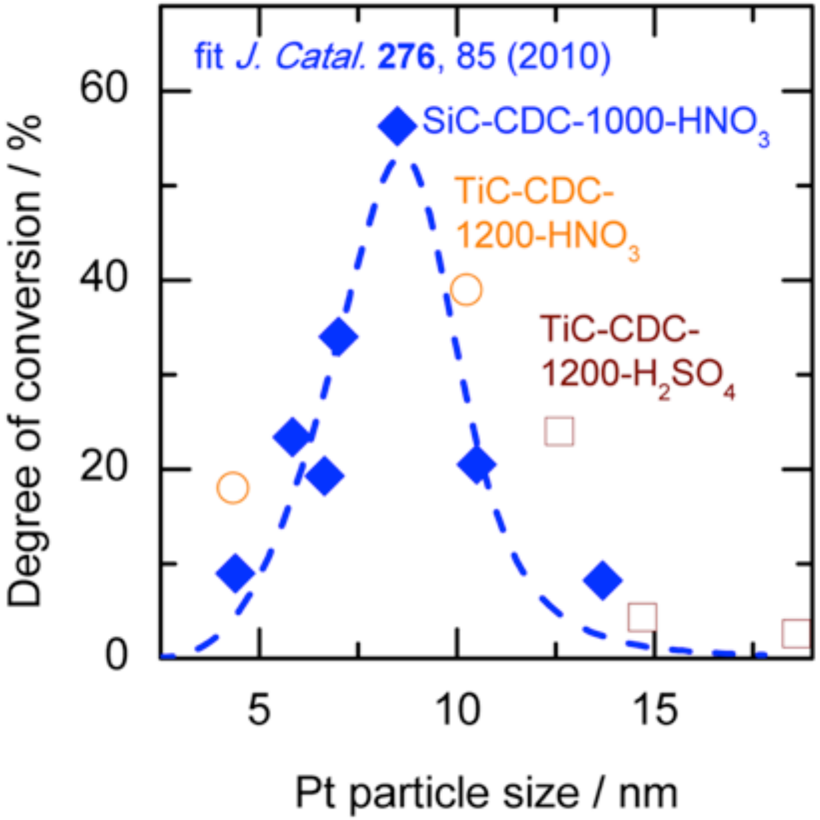
fragrance / flavor

cooling effect

anesthetic

Etzold et. al. *Catalysis today* **140**, 30-36 (2009)

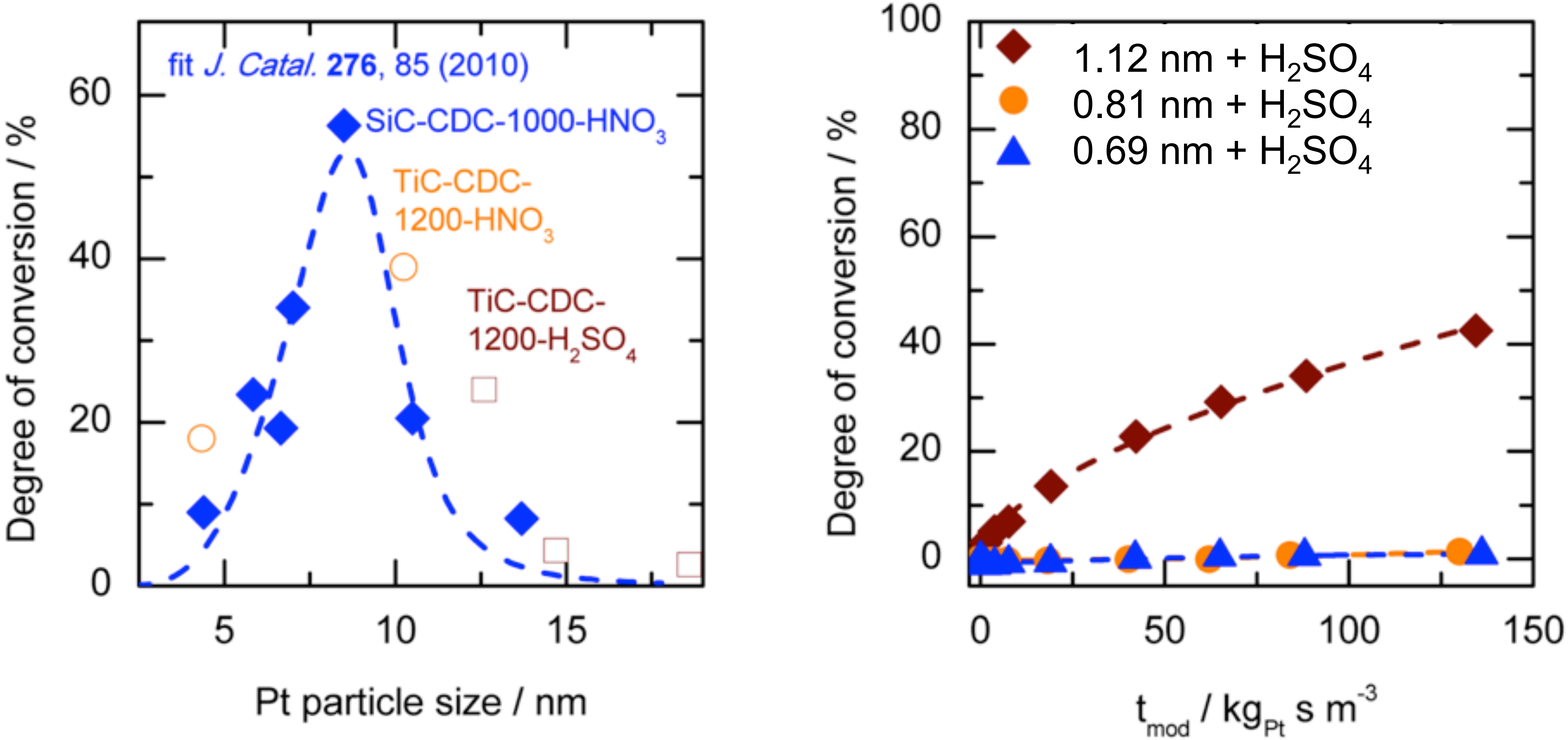
Application of carbonaceous catalysts: Menthol



5 mg thymol, 150 ml cyclohexan, 2.5 wt.-% Pt, 150 °C, 5 MPa H_2, 600 min^{-1}

- Structure sensitive reaction

Application of carbonaceous catalysts: Menthol

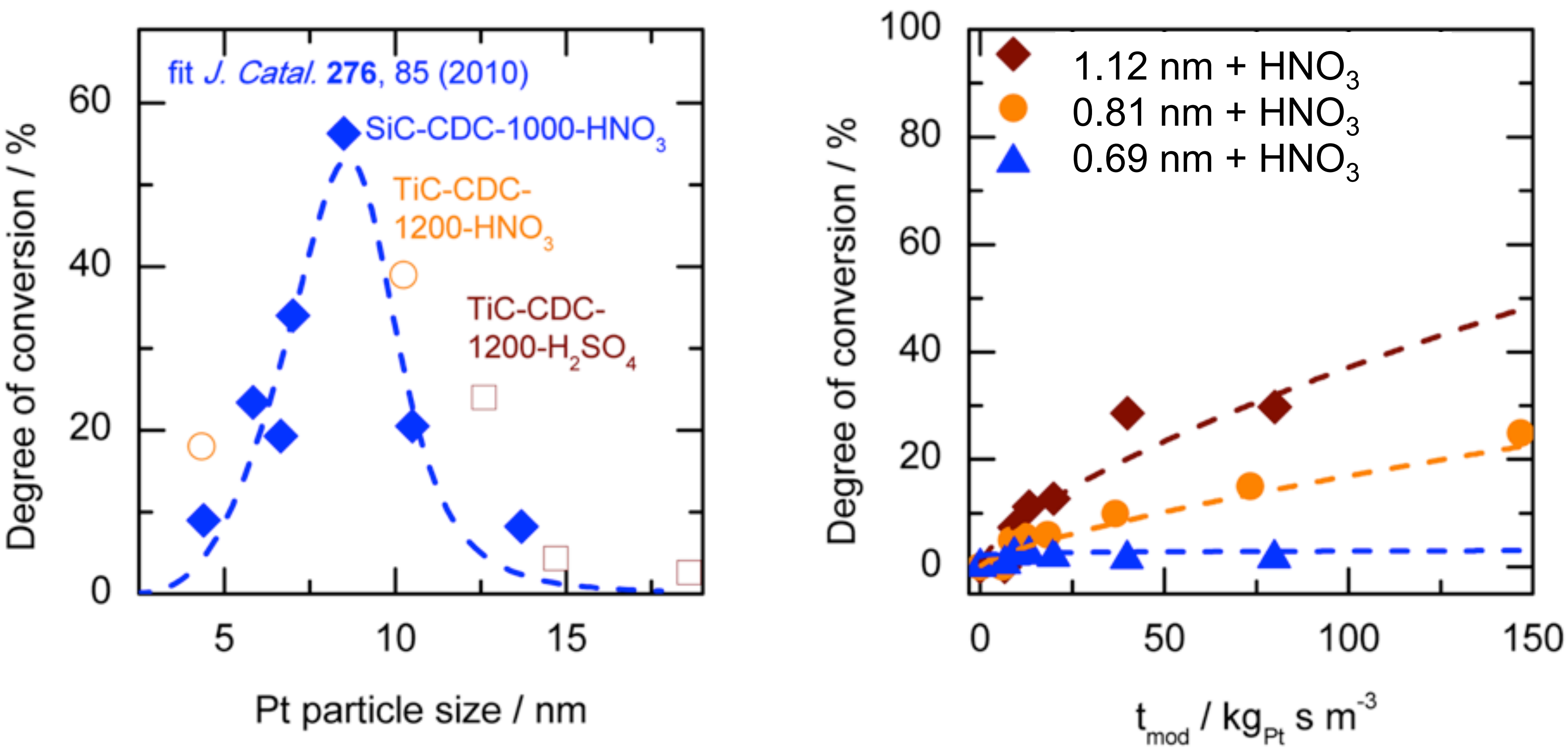


5 mg thymol, 150 ml cyclohexan, 2.5 wt.-% Pt, 150 °C, 5 MPa H_2, 600 min^{-1}

- Structure sensitive reaction
- Molecular sieve character induces shape selectivity

Glenk et al. *Chem. Eng. Technol.* **33**, 698 (2010); Silvestre-Albero et al. *Carbon* **59**, 221 (2013)

Application of carbonaceous catalysts: Menthol

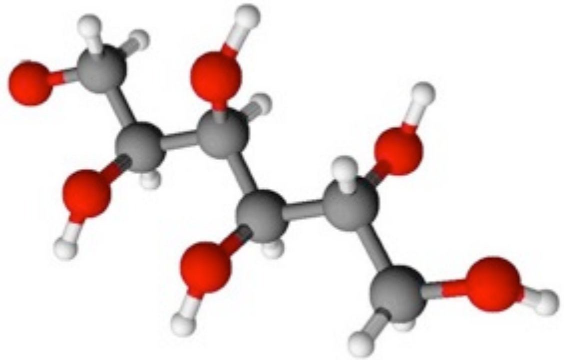


5 mg thymol, 150 ml cyclohexan, 2.5 wt.-% Pt, 150 °C, 5 MPa H_2, 600 min^{-1}

- HNO_3 oxidation influences molecular sieve character

Glenk et al. *Chem. Eng. Technol.* **33**, 698 (2010); Silvestre-Albero et al. *Carbon* **59**, 221 (2013)

Aqueous Phase Reforming (APR)



H_2

Hydrocarbons

alcohols, polyols

CO $\xrightarrow{H_2O}$ $CO_2 + H_2$

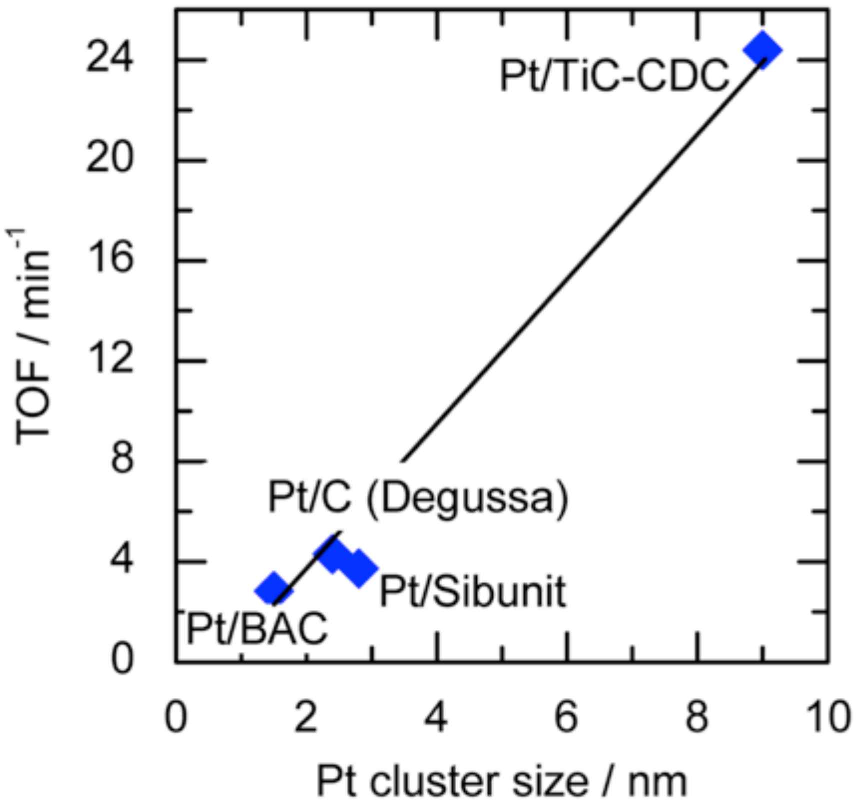
210-250°C High pressure (20-50 bar) Liquid water

→ Hydrothermal stable supports → Carbon

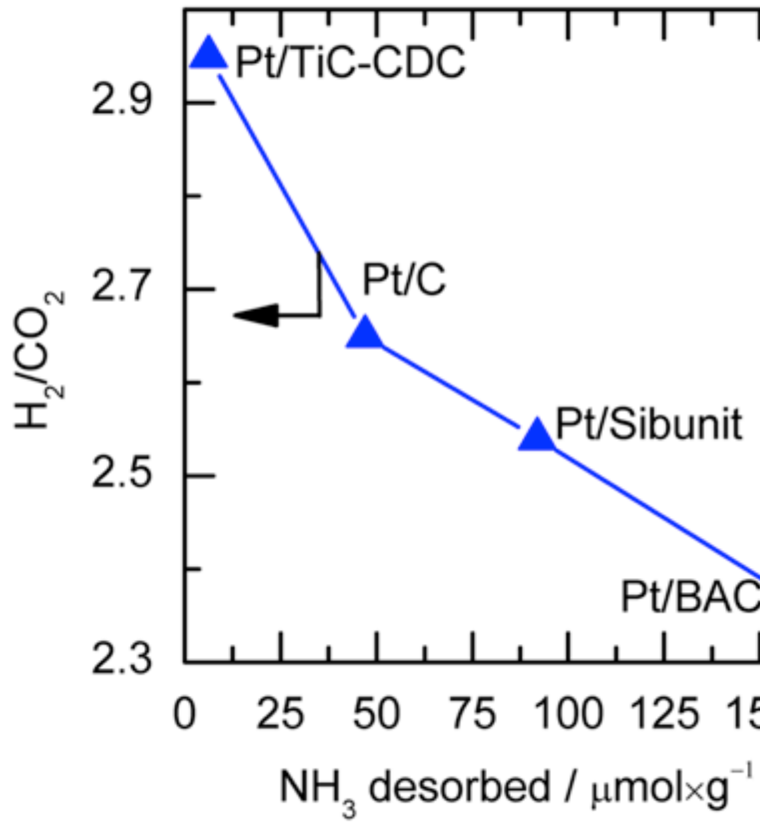
Huber et al. *Science* **300** 2075 (2003); Ravenelle et. al. *ACS Catal.* **1** 552 (2011)

APR: activity & selectiviy

Activity



H_2 selectivity



xylitol, 498 K, 29.3 bar, N_2 flow 30 ml min^{-1}, WHSV = 3.0 h^{-1}.

- Face atoms seem to be more active than corner and edges
- Acidity allows to tune H_2 and alkane selectivity

Kirilin et. al. *Catal. Sci. Technol* **3**, 387 (2014)

Tuning carbon catalysts properties matters!

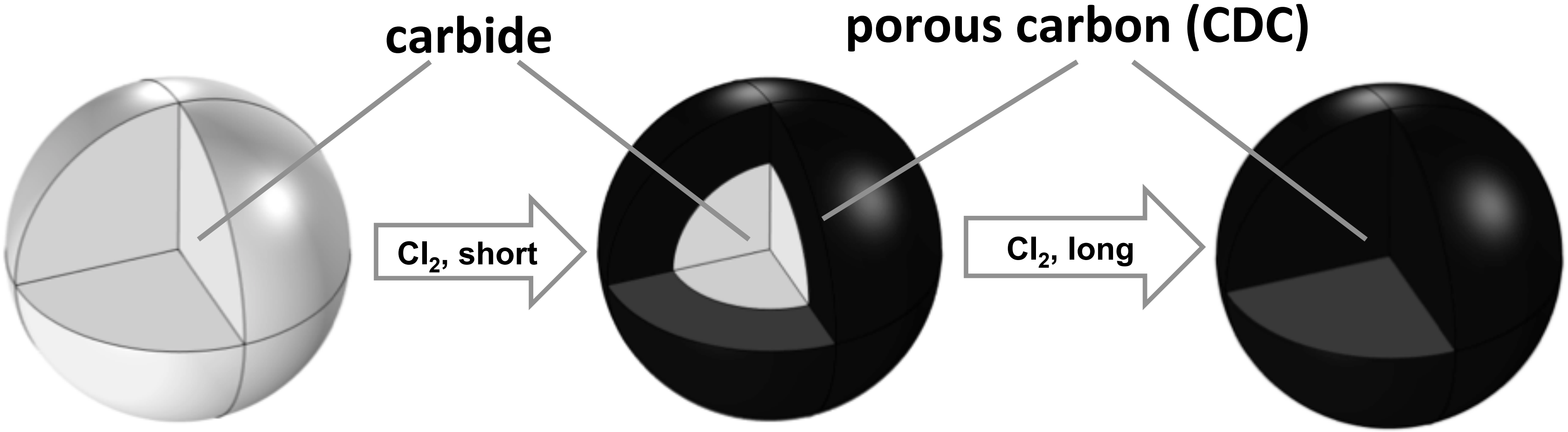
Some results on how to precisely control carbonaceous materials properties

Drawbacks of activated carbon from natual feedstock

- Broad fluctuation in material properties
- Uncontrolled pore size distributions
- High amount of impurities (ash content)
- Varying surface functionalization

- Using more reliable feedstock mitigates this problems.
  Alternatives are: Polymers and Carbides

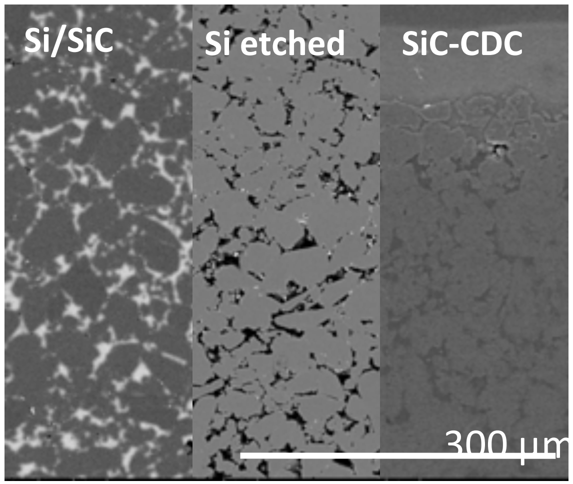
Carbide-derived carbon



$$SiC(s) + 2Cl_2(g) \rightarrow SiCl_4(g) + C(s)$$

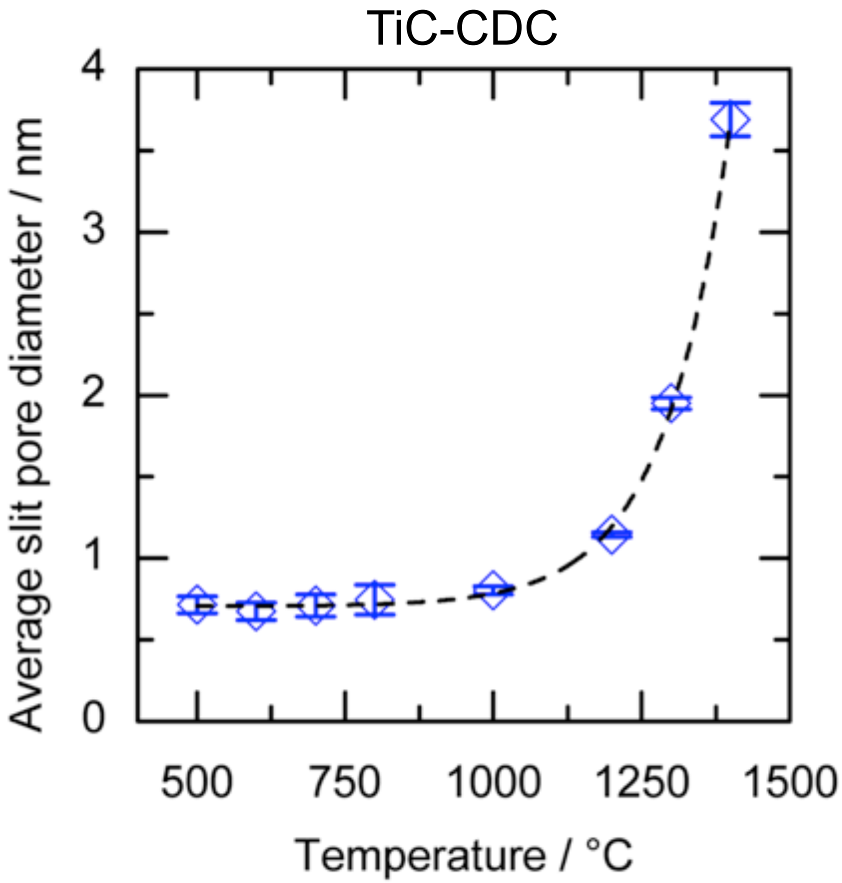
$$TiC(s) + 2Cl_2(g) \rightarrow TiCl_4(g) + C(s)$$

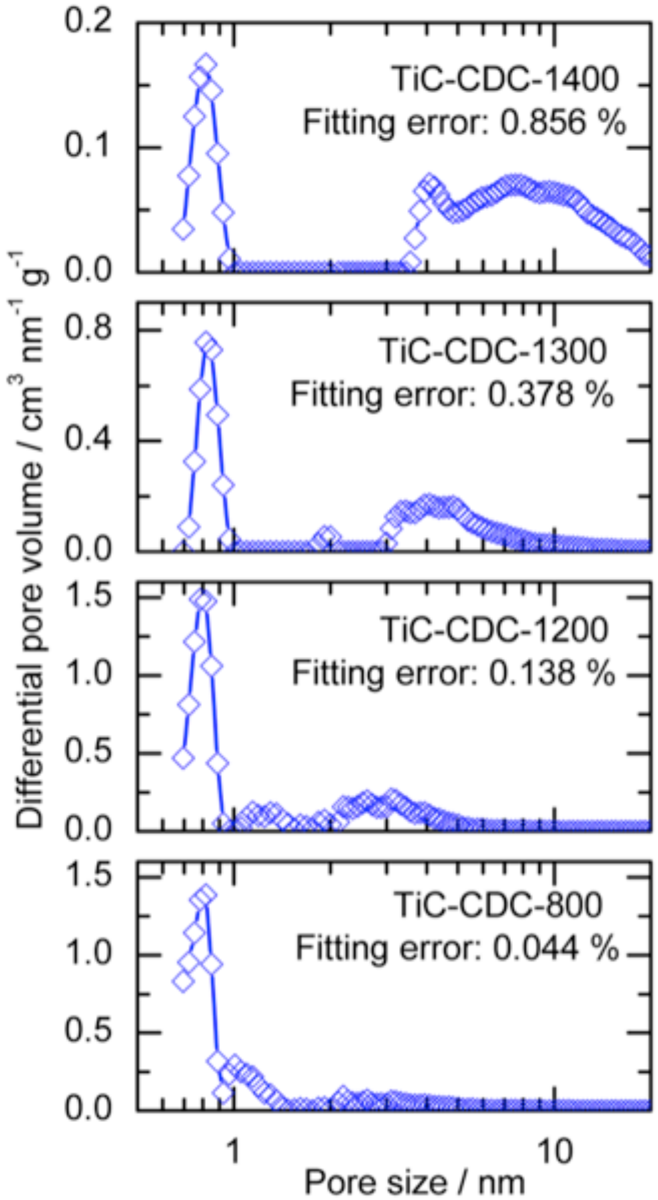
Review: V. Presser, M. Heon, Y. Gogotsi, *Adv. Funct. Mater.* 21, p. 810 (2011).



Etzold et. al, *Carbon* 49 (2011) 4359-4367

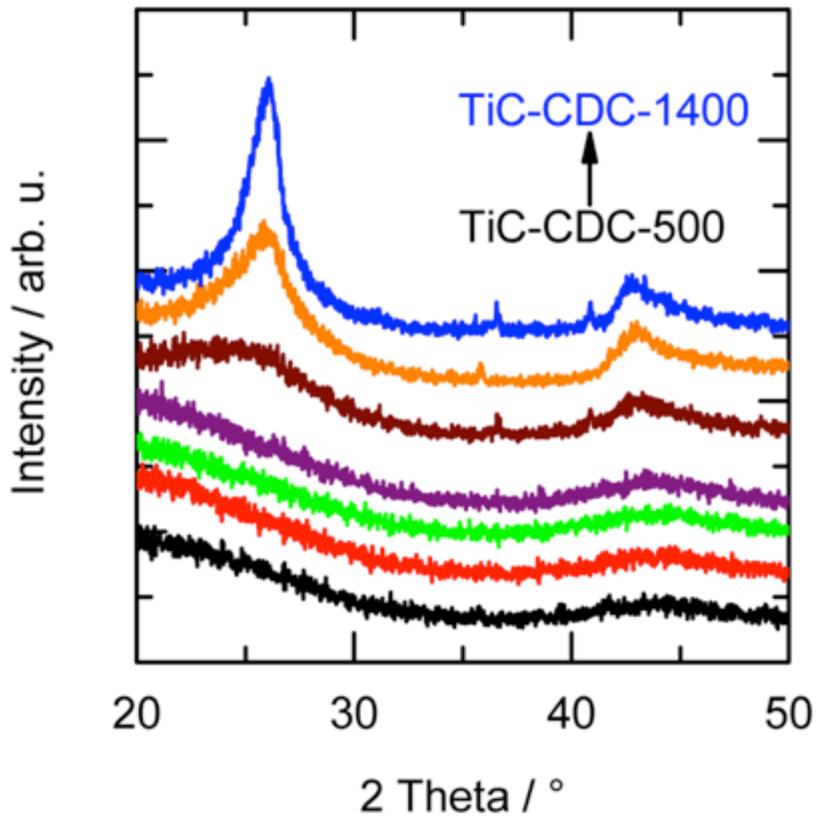
CDC: Pore size control

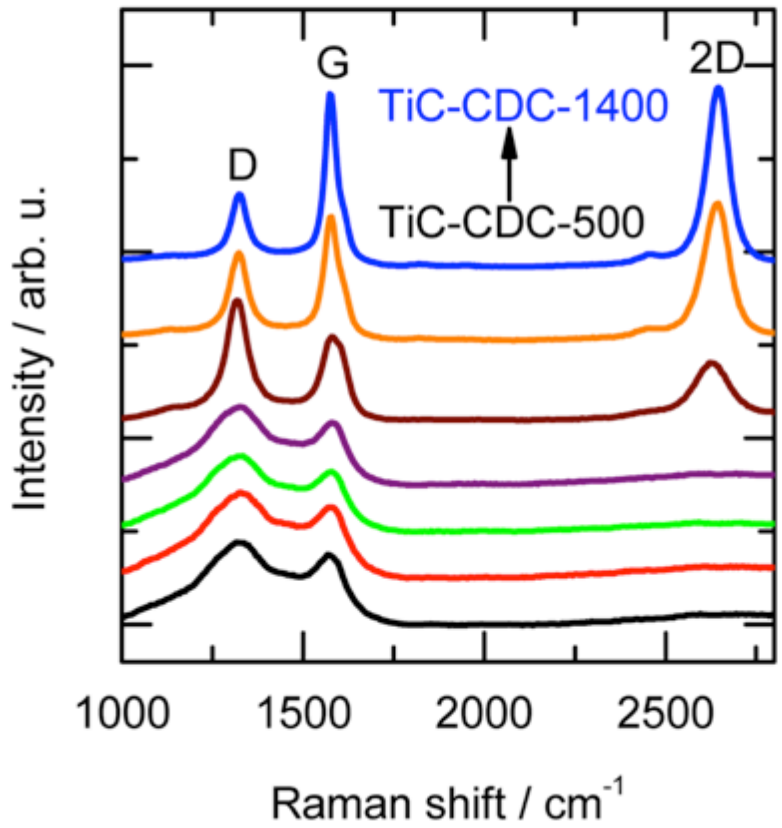




- High control of pore size distribution in the micro- & macropore regime

CDC: microstructure

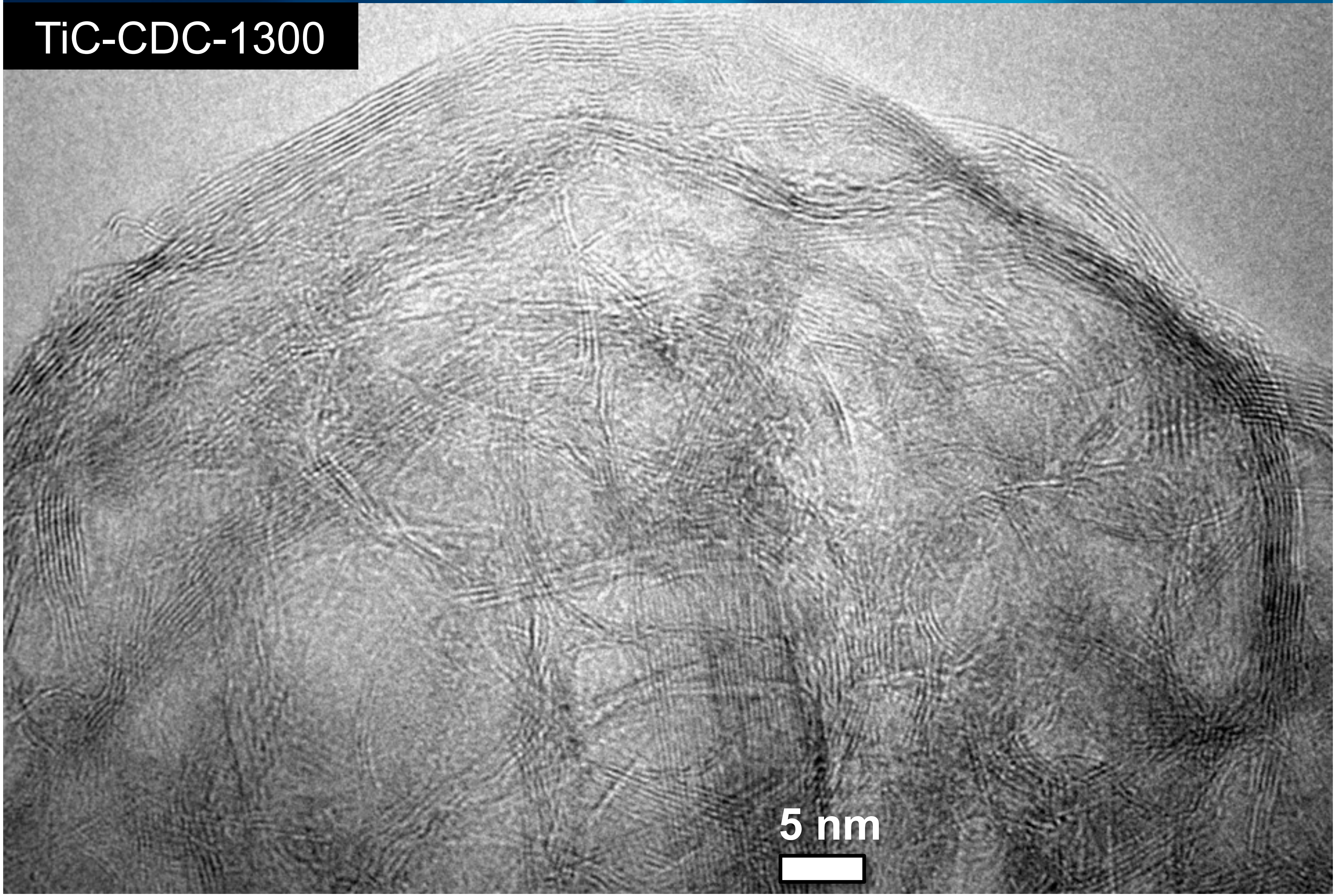




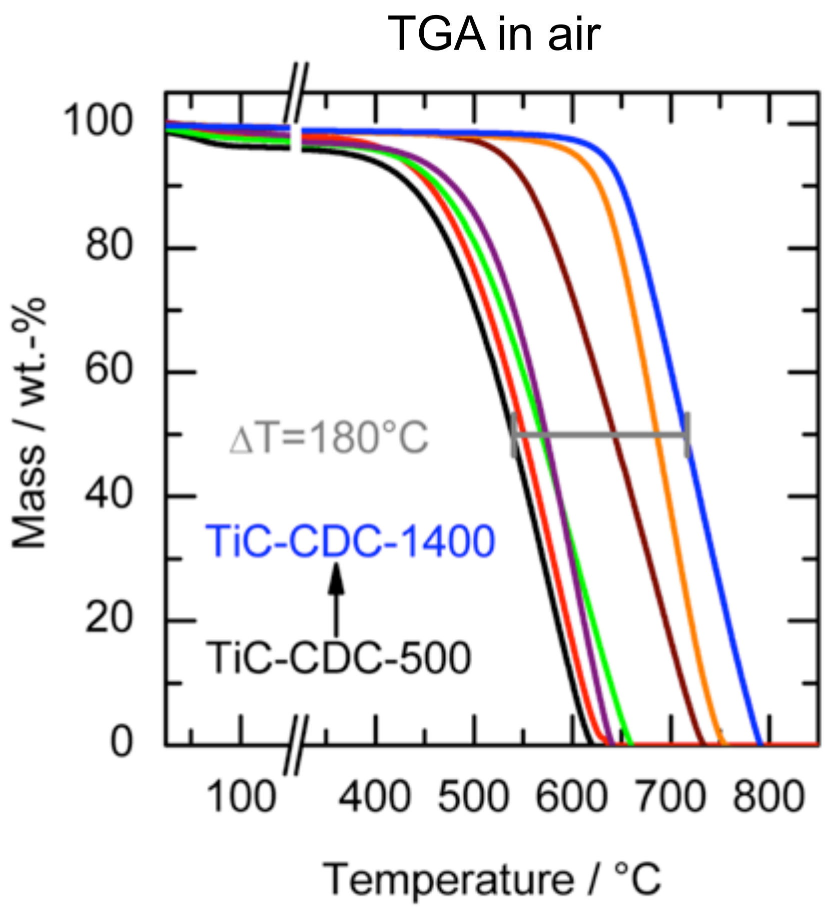
Reason for pore size control:

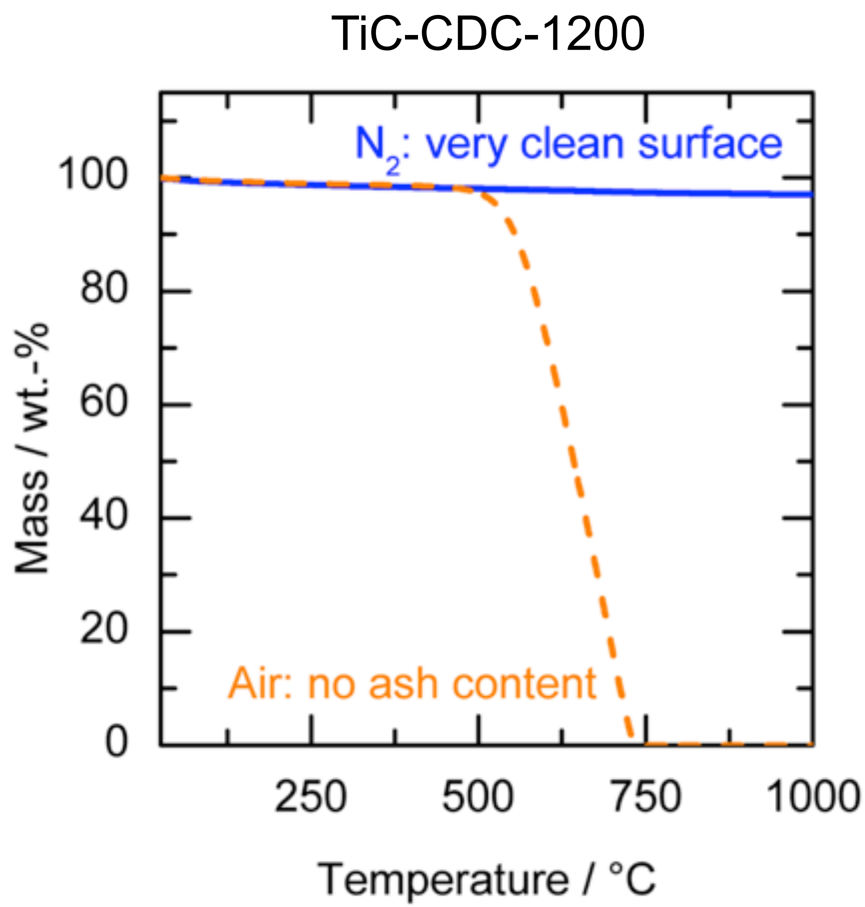
- Higher degree of graphitization with increasing temperature
- Graphitic ribbons build mesopores

CDC: microstructure



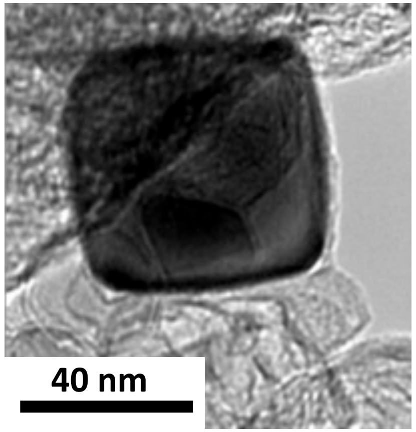
CDC: purity

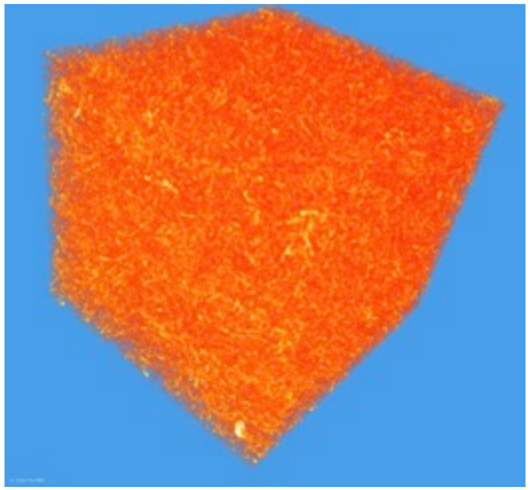




- Strong influence of microstructure on chemical stability
- No ash content → high purity
- No surface groups desorbing → very clean surface

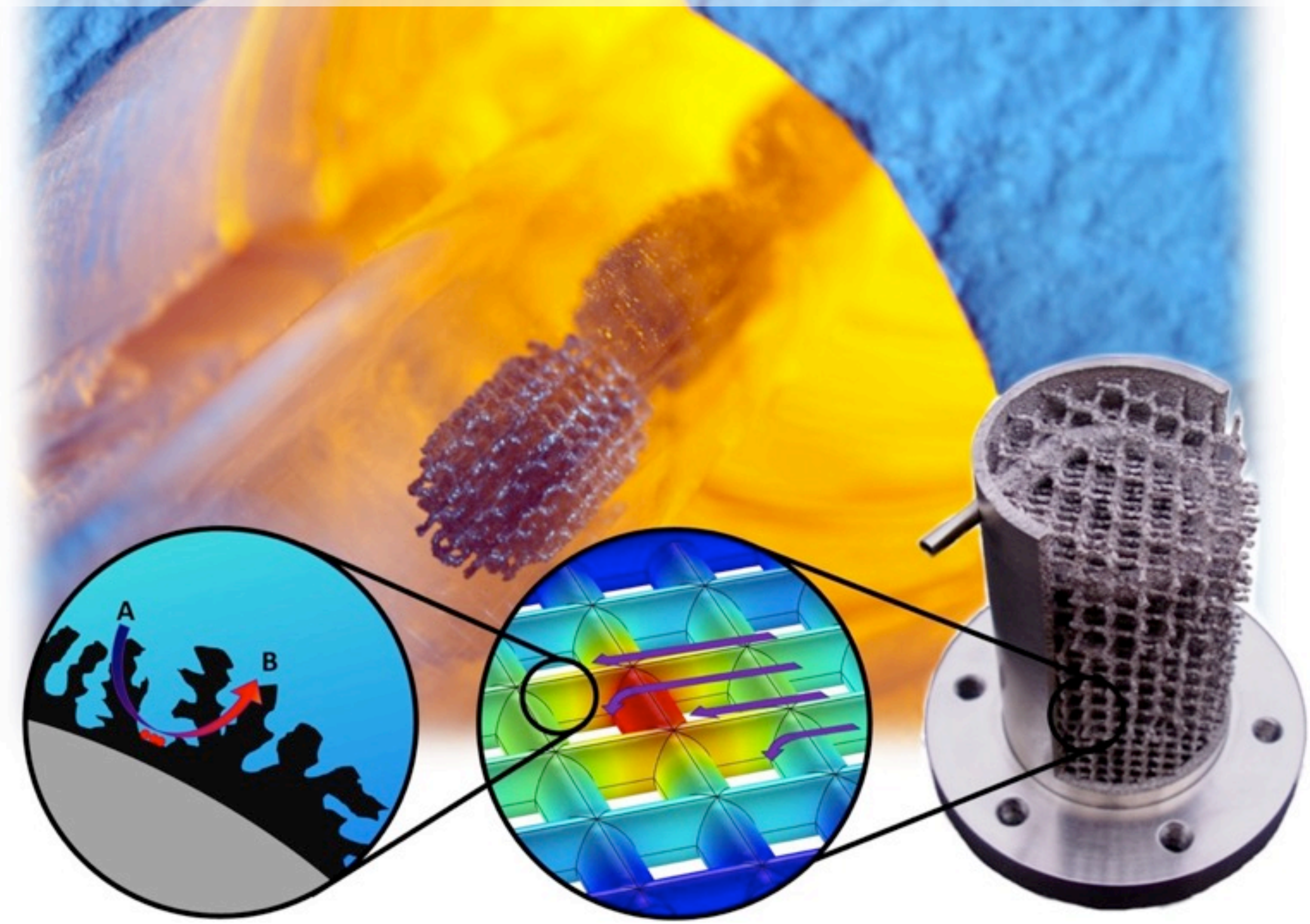
From nano to macro to structured ....



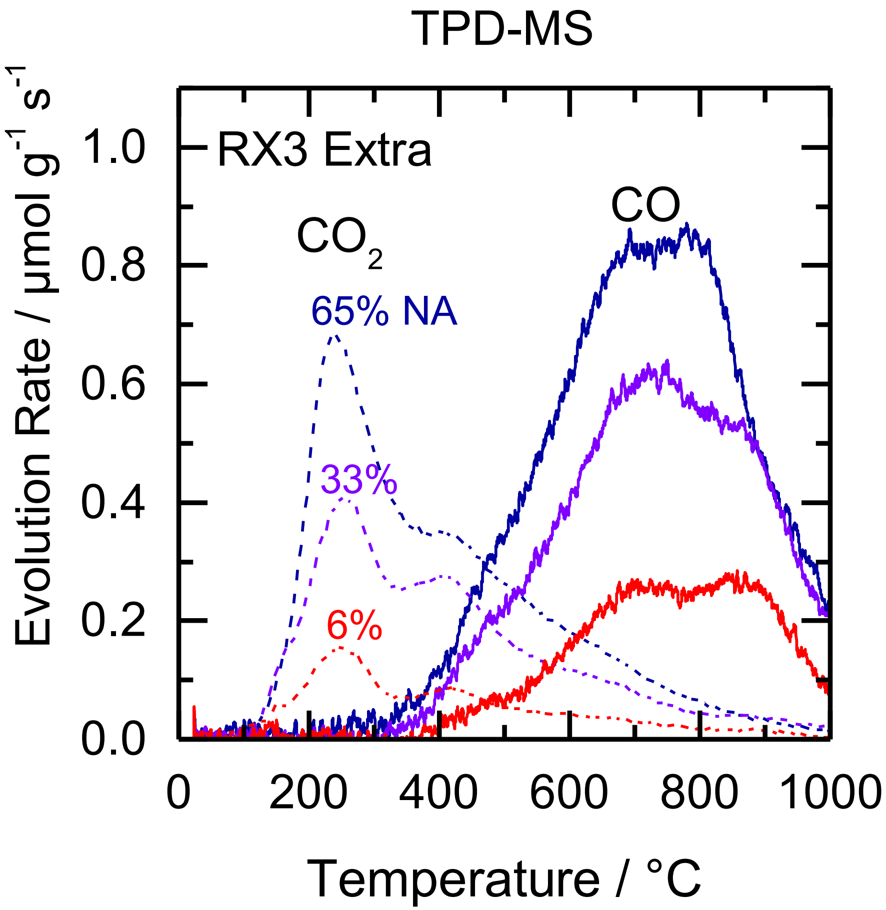


... and coatings

Front Cover: *Chem. Eng. J.* **181-182** (2012)



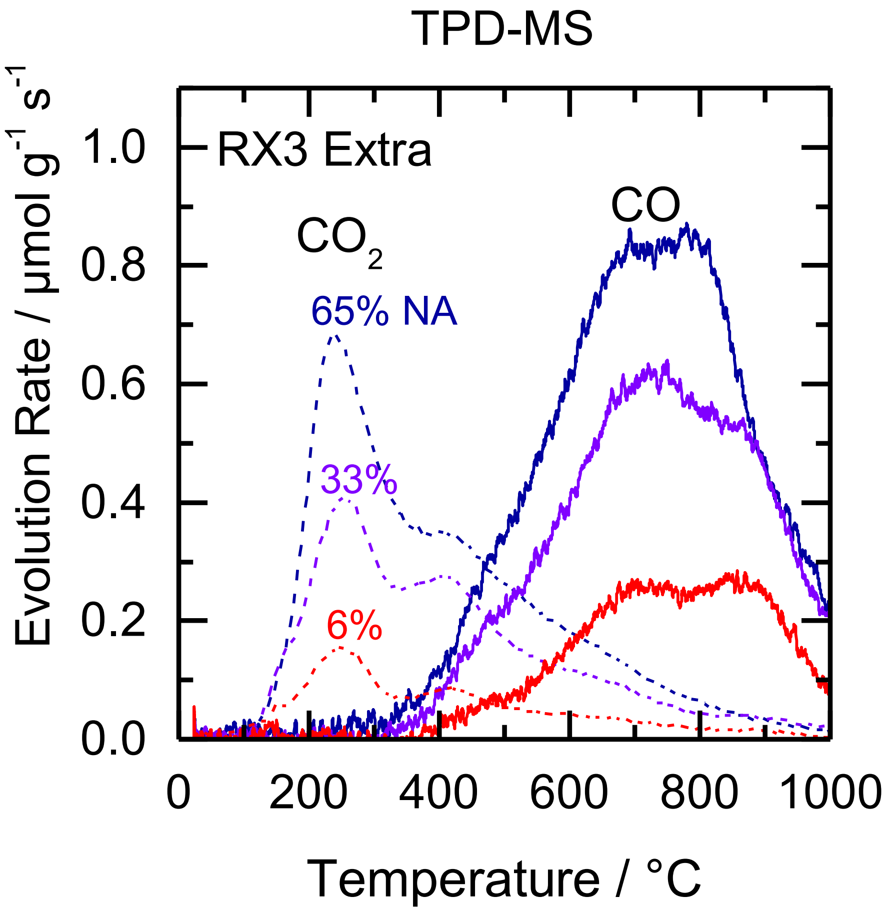
Introducing oxygen groups

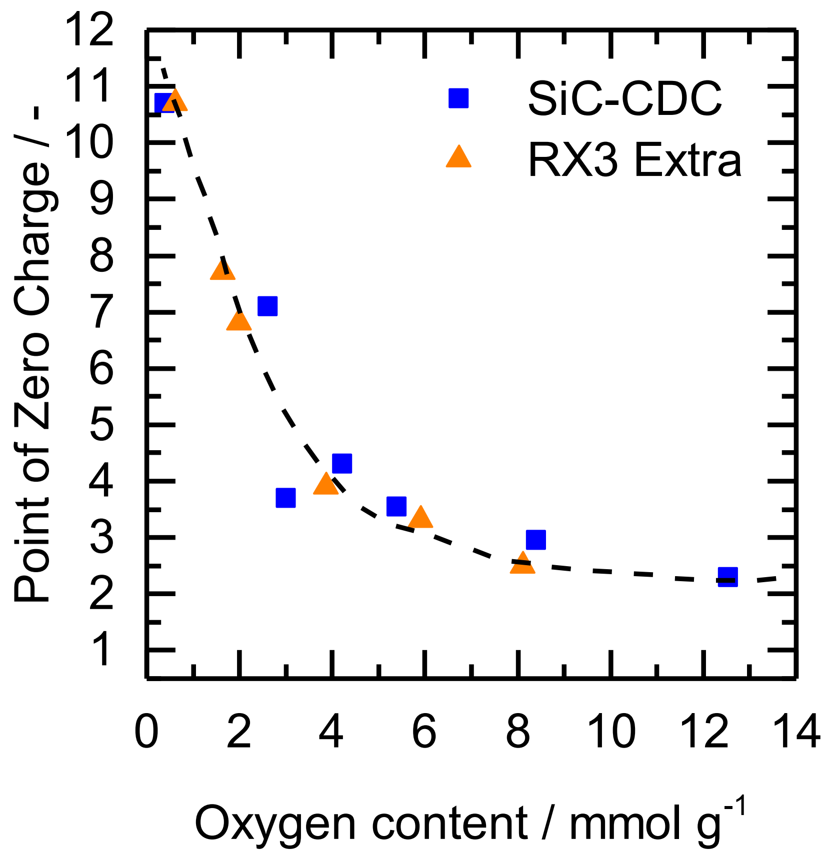


- The pristine carbon surface can be functionalized through partial oxidation with various gas or liquid phase reactants (H_2SO_4, HNO_3, H_2O_2, O_2, O_3).
- A variation of nitric acid concentration is given in the left figure.

- Amount of surface groups can be altered with oxidation agent, concentration, temperature, …

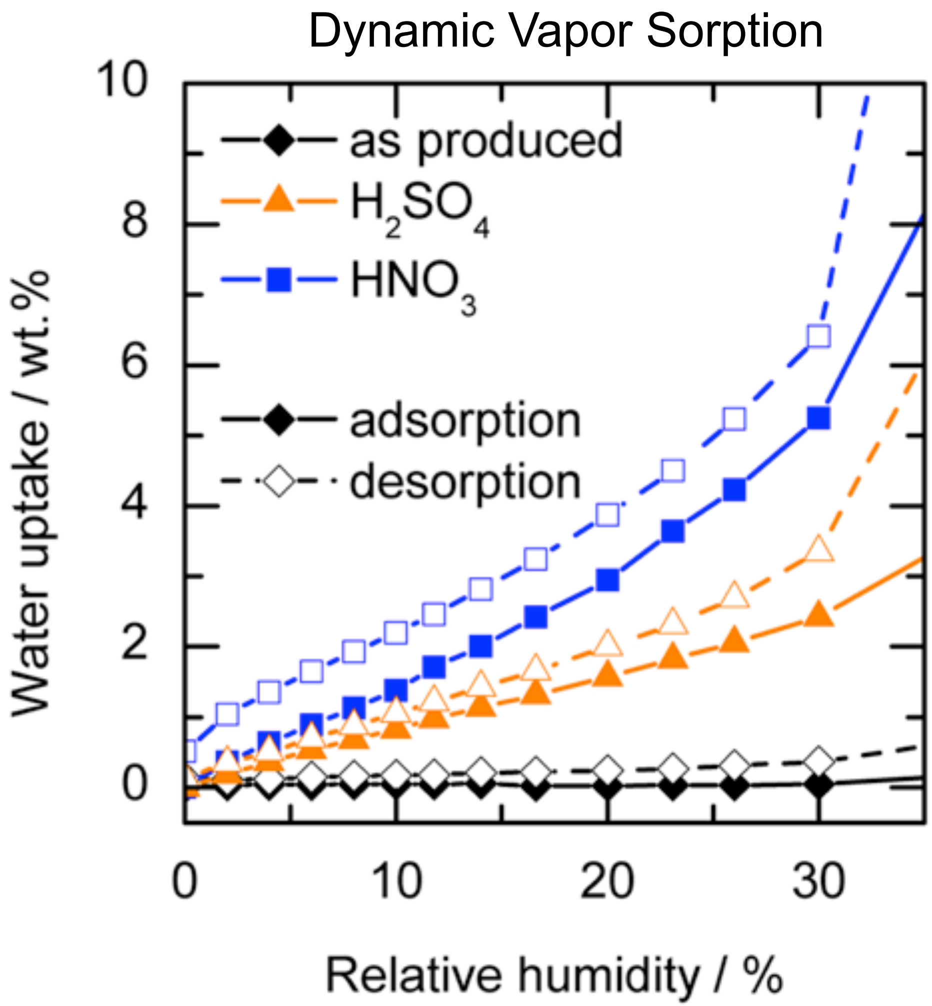
Introducing oxygen groups – dependency of PZC





- Amount of surface groups can be altered with oxidation agent, concentration, temperature, …
- Decrease of PZC till approx. 4 mmol g^{-1} g oxygen.

Introducing oxygen groups – water interaction



- Highly pure carbon → hydrophilic
- Introducing surface oxygen breaks hydrophobic character

DVS, 40.2 °C, 10 mg, pre drying 120 °C, 1h

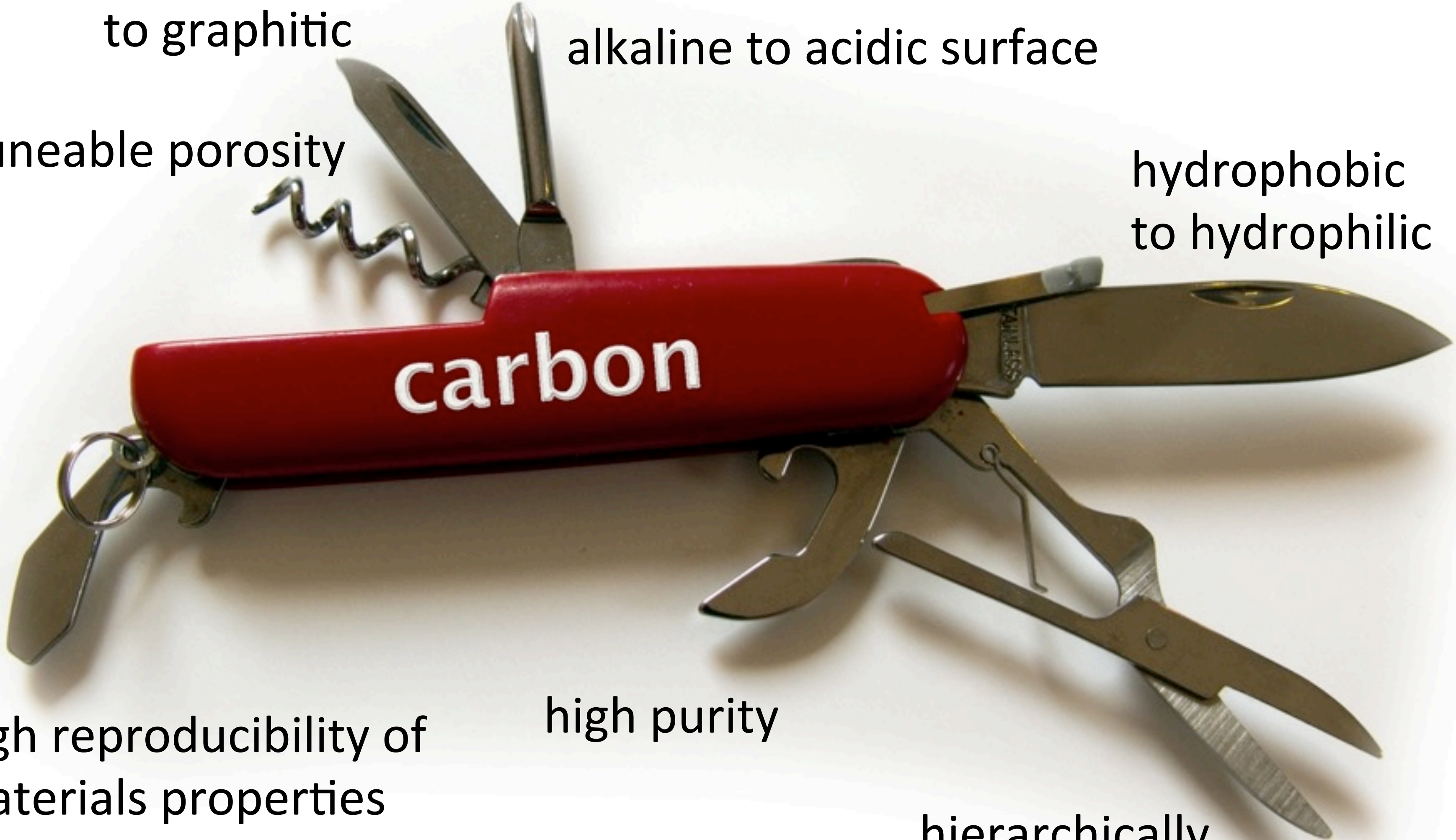
Materials toolbox

amorphous
to graphitic

alkaline to acidic surface

tuneable porosity

hydrophobic
to hydrophilic



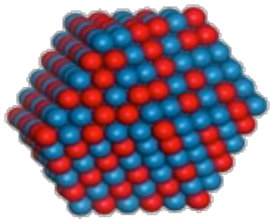
high reproducibility of
materials properties

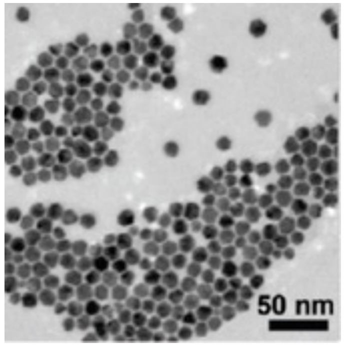
high purity

hierarchically
structured

Deposition and tuning of active sites

- *Ex situ* nanoparticle synthesis



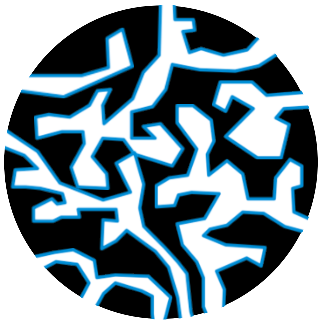


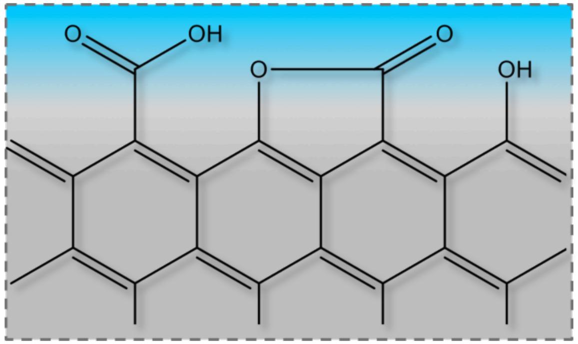
- Ion adsorption
  - good control of loading
  - good control of metal cluster size
  - needs functionalized surface

- Incipient wetness impregnation
  - works on pristine carbon surfaces

Ion adsorption technique

Porous carbon with oxidized surface



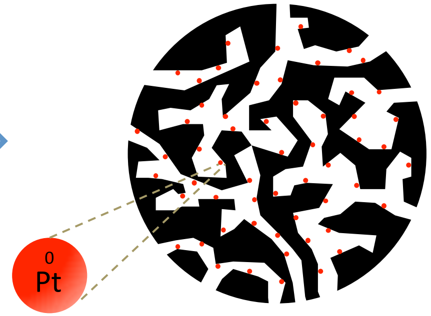


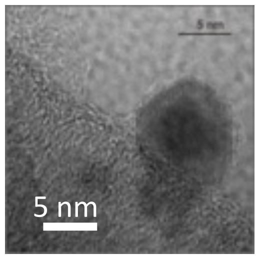
Ion adsorption of metal salts

$[PtCl_6]^{2-}$

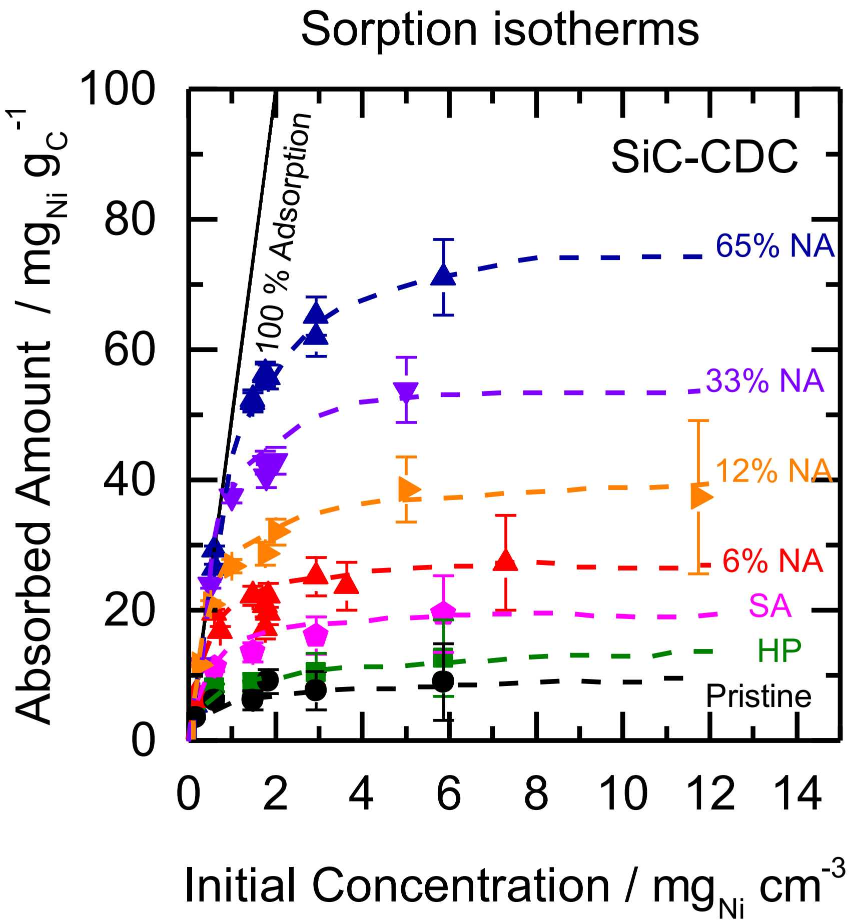
$[Pt(NH_3)_4]^{2+}$

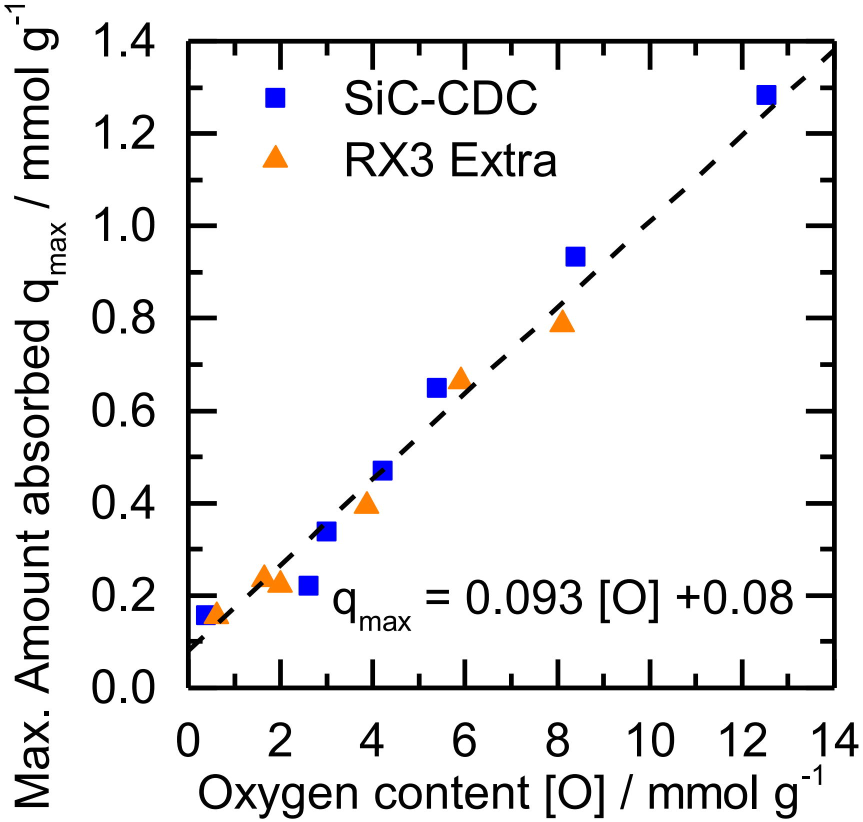
Wet chemical or gas phase reduction





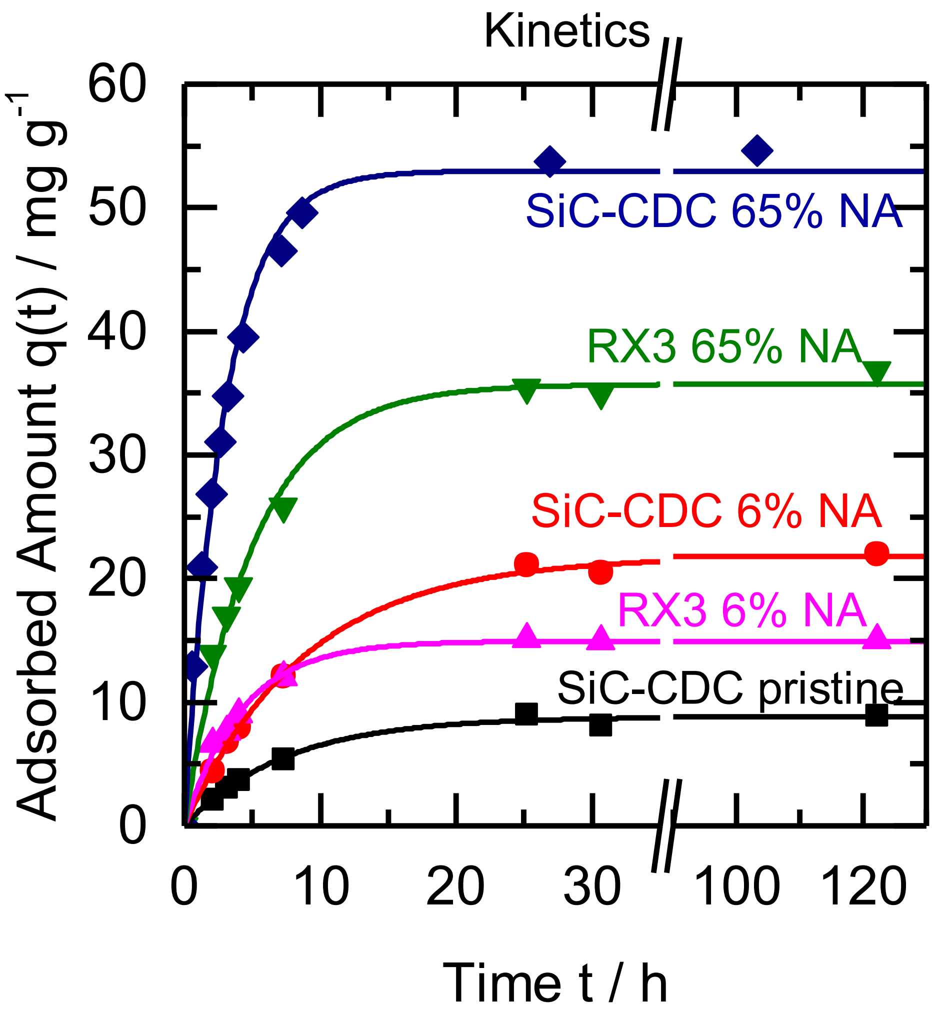
Ion adsorption: surface groups & loading (Ni)





- For low concentrations full adsorption is achieved → control loading
- Oxygen content determines maximum loading

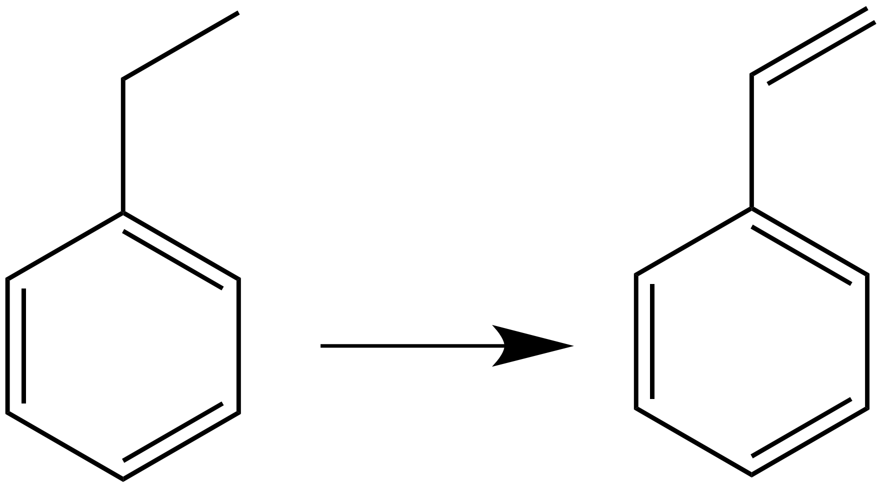
Ion adsorption: adsorption time & loading (Ni)

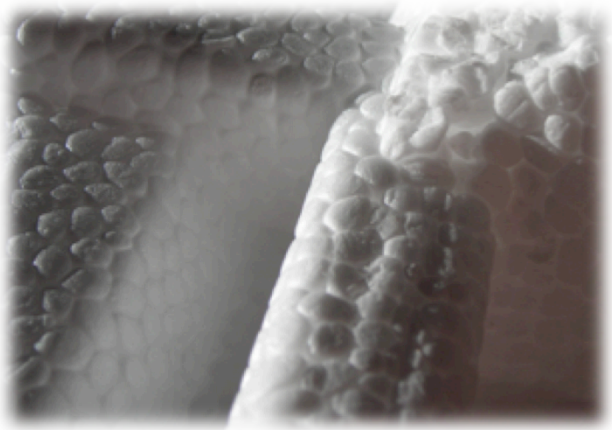


- Kinetics of adsorption need to be accounted
- Rate constant is similar for different oxidative pre-treatments
- Approx. 10 h of adsorption are sufficient

Two unconventional examples and success stories
with carbon in catalysis
at the end.

Direct dehydrogenation of ethylbenzene

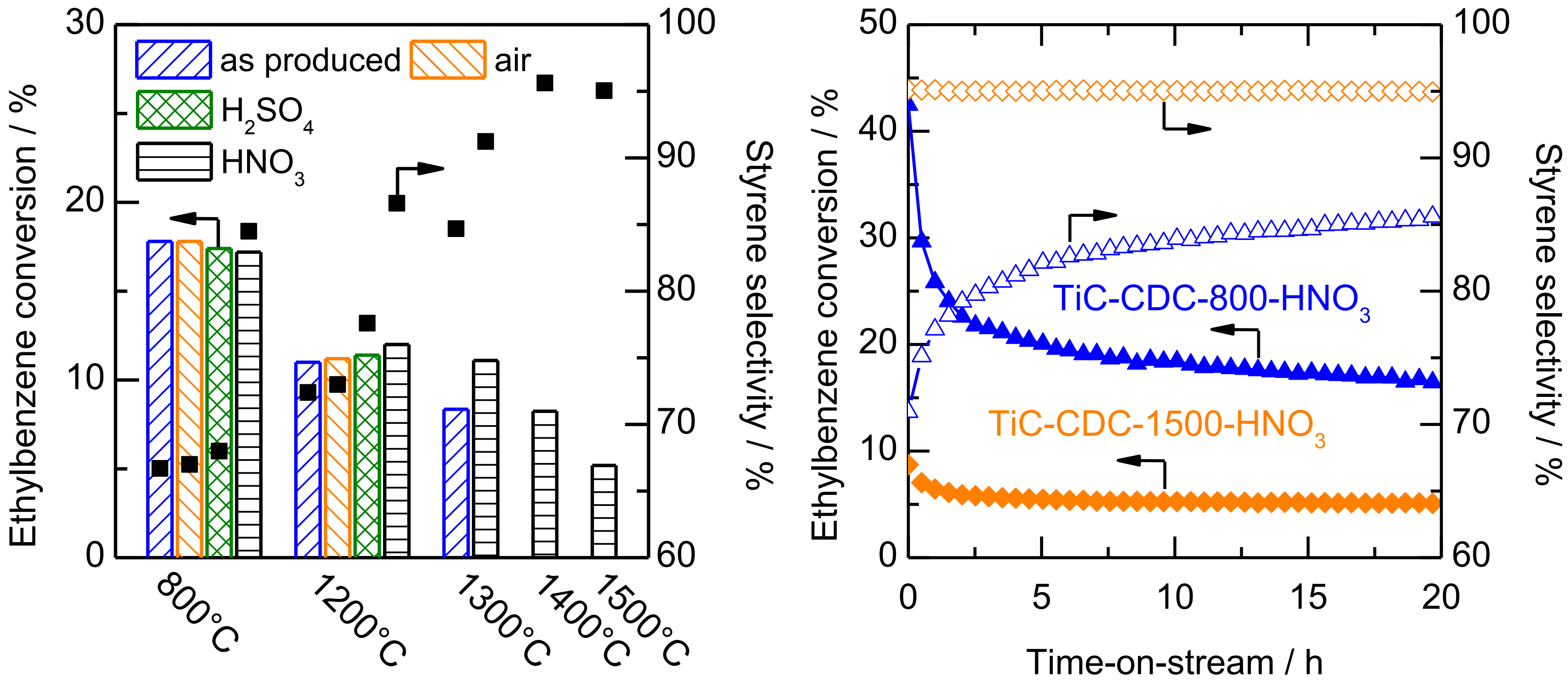




- Carbon (without active metal) catalyses the styrene production in a Mars-van-Krevelen type mechanism.

Zhang et al. *Angew. Chem. Int. Ed.* **49**, 8640 (2012)

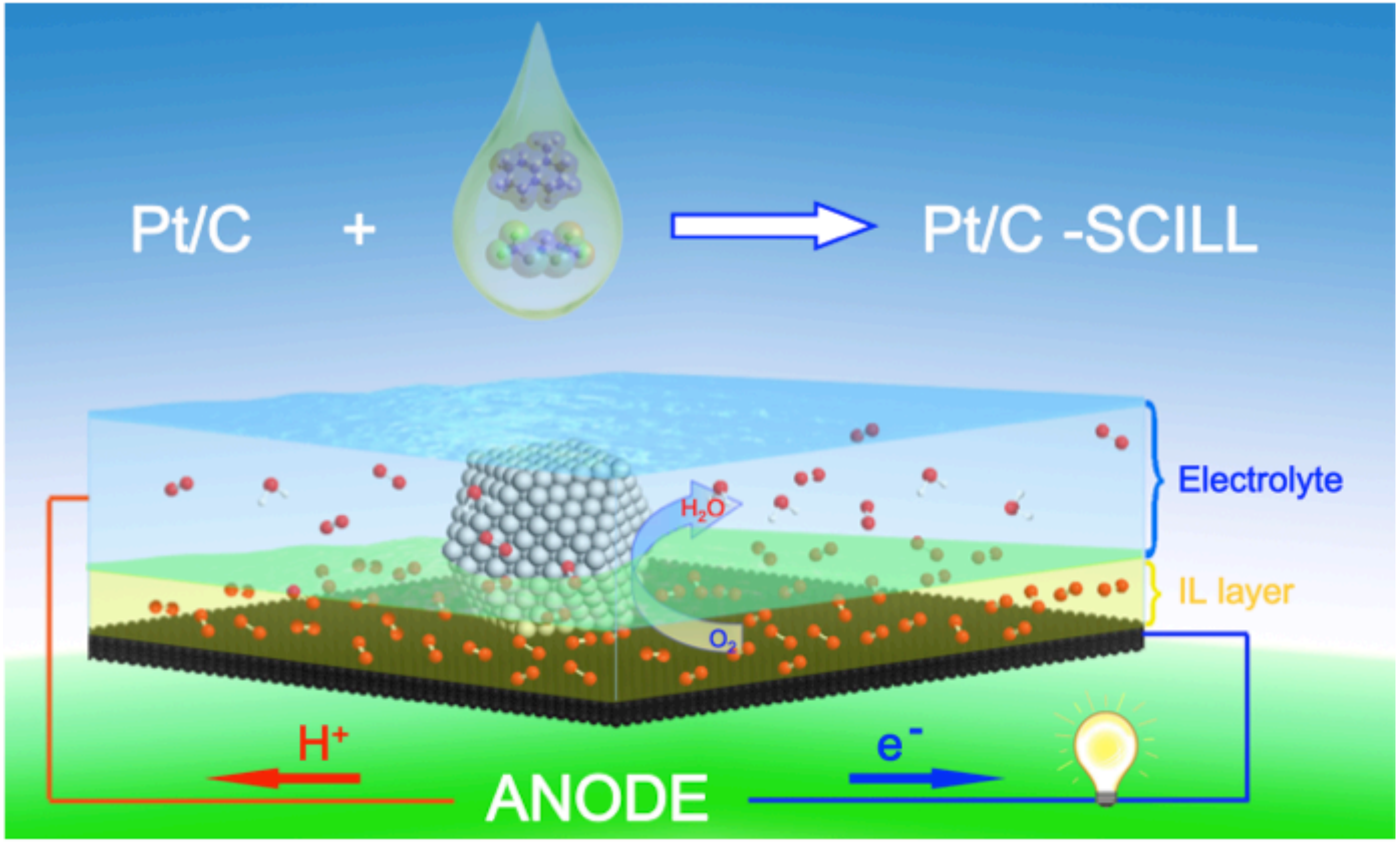
Direct dehydrogenation of ethylbenzene with TiC-CDC



100 mg carbon, 550 °C, WHSV = 6000 h^{-1}

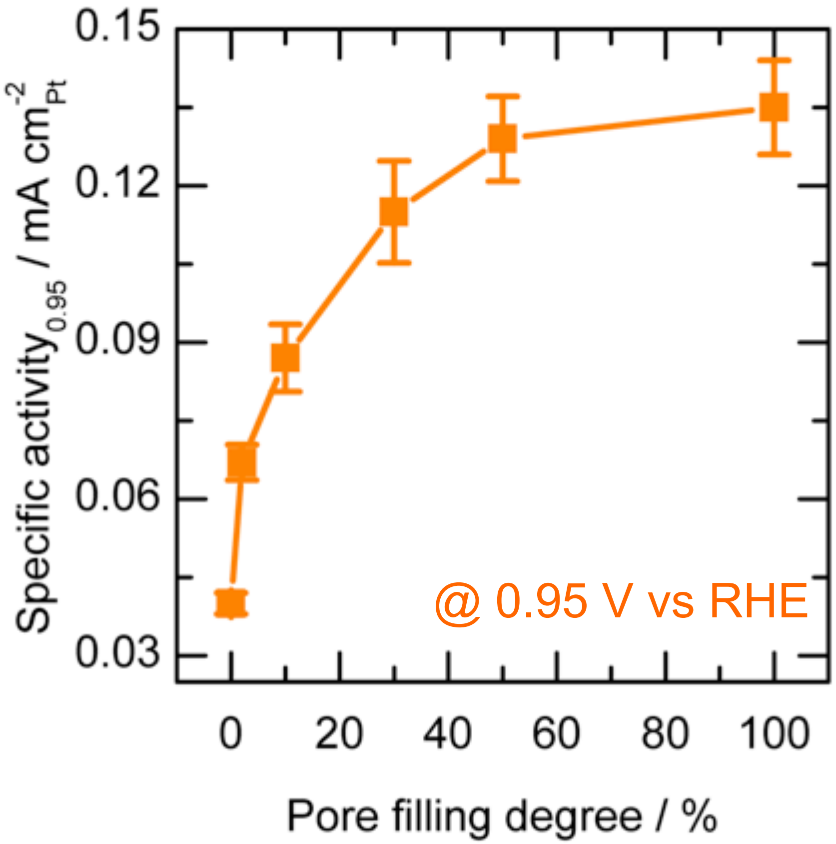
- HNO_3 oxidized high temperature TiC-CDC shows high selectivity
- Stable catalysts after induction period

Ionic Liquid modification of carbon supported electrocatalysts

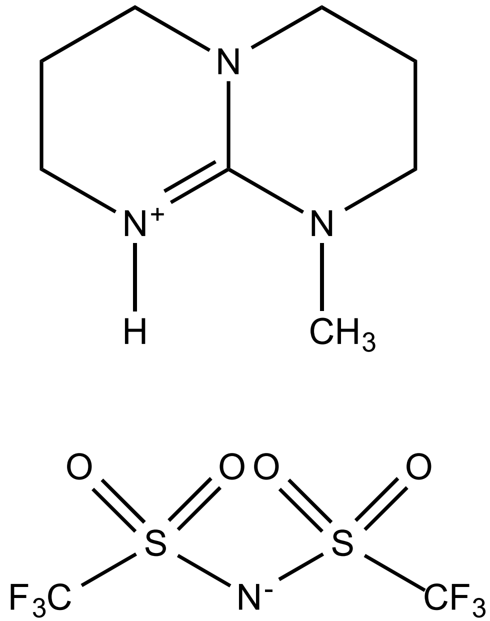


Kernchen et al. *Chem. Eng. Technol.* **30**, 985 (2007)
Zhang et al. *ACS Appl. Mater. Interfaces* ***18**, 3562-3570 (2015)*

Influence on ORR activity



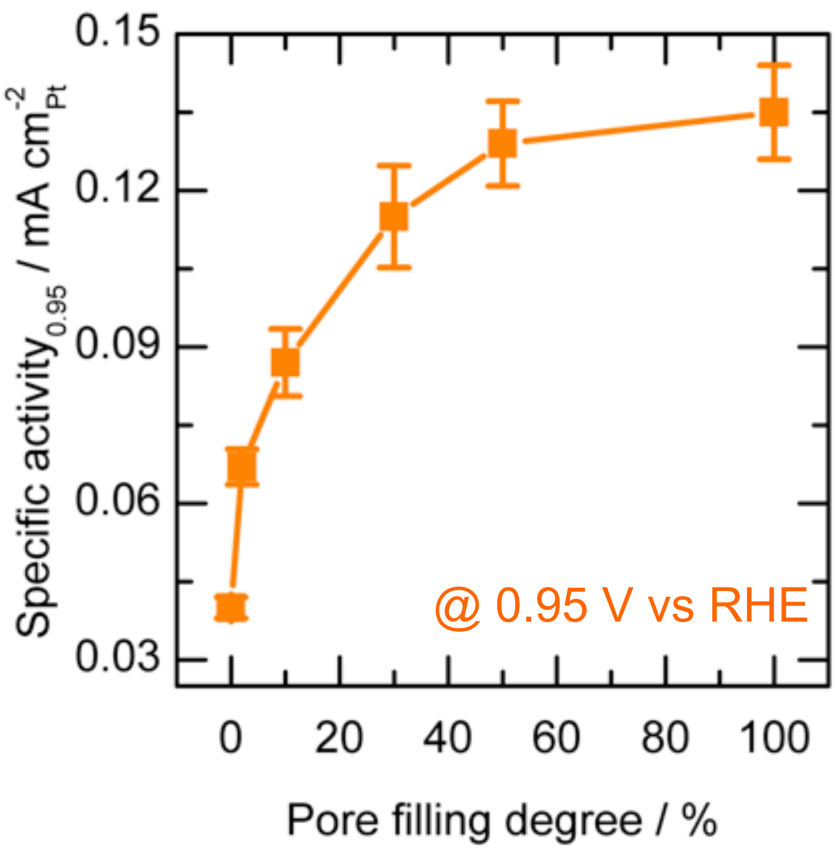
$[MTBD][NTf_2]$

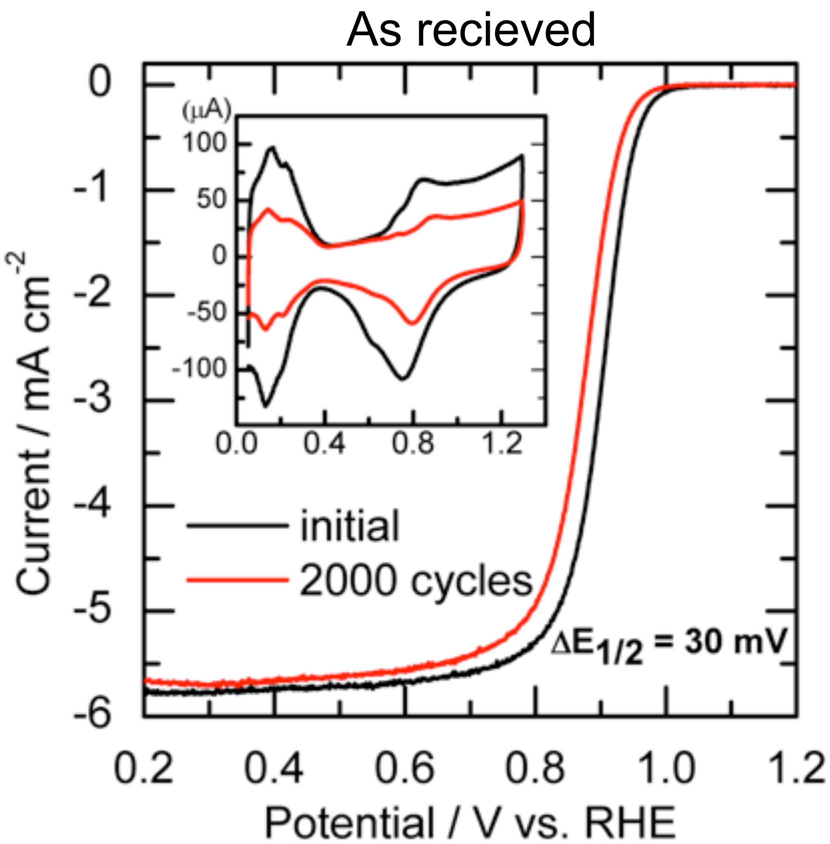


0.1 M $HClO_4$, 10 mV s-1, 1600 rpm, room temperature, $[MTBD][N(SO_2CF_3)_2]$

- Pronounced increase in activity by a factor of **3.4**.
- Effects are: solubility, ligand effect, blocking of defects

Influence on ORR stability

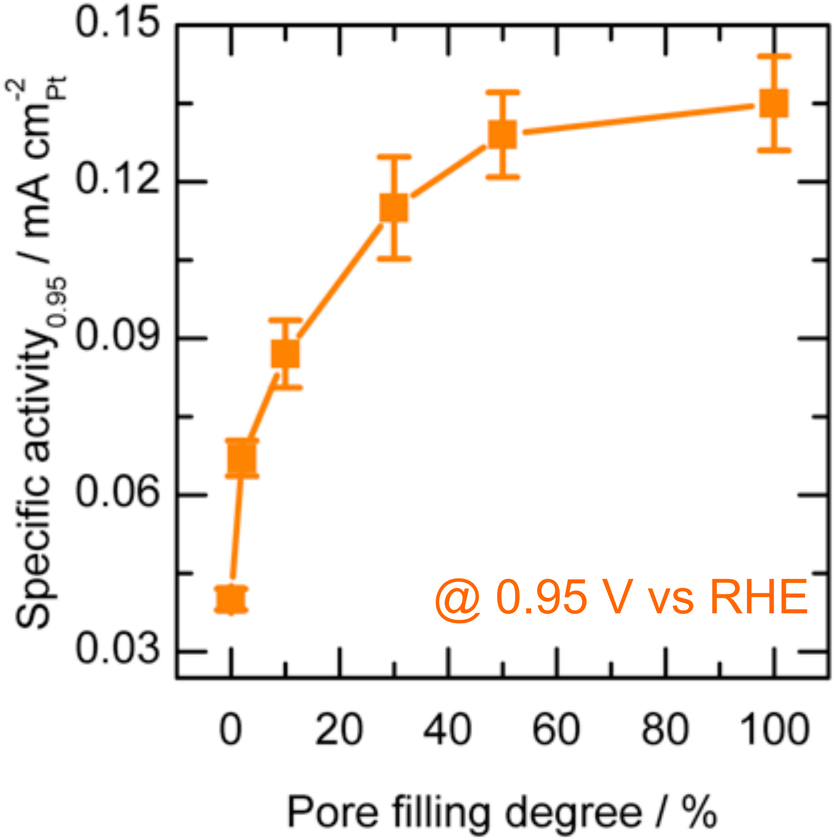


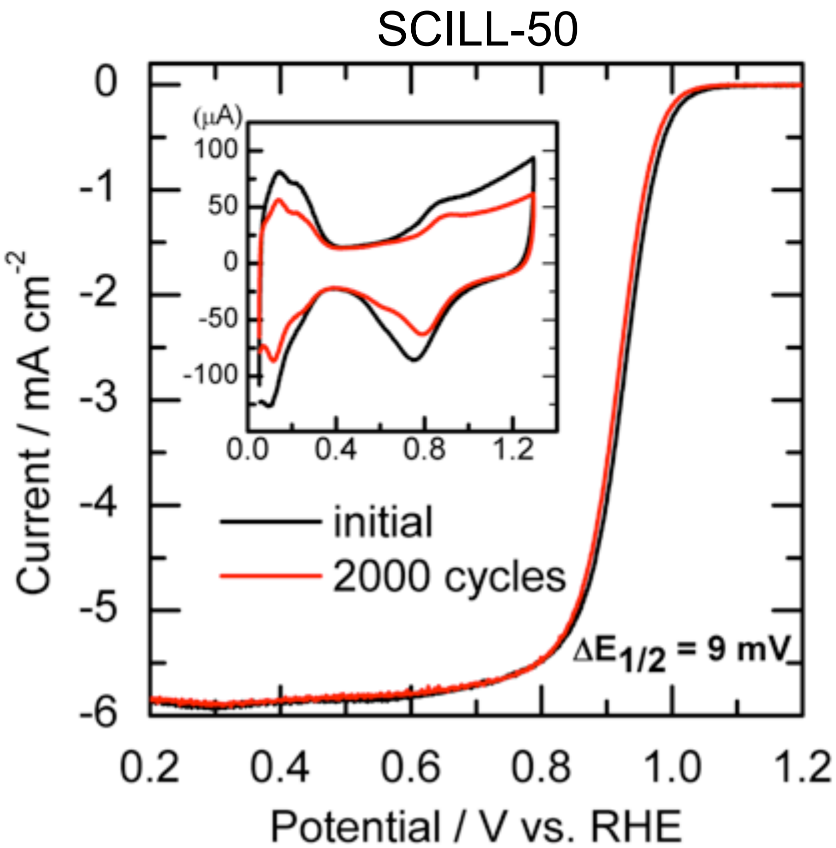


0.1 M $HClO_4$, 10 mV s-1, 1600 rpm, room temperature, [MTBD][$N(SO_2CF_3)_2$]

- Non modified catalyst degrades strongly.

Influence on ORR stability





0.1 M $HClO_4$, 10 mV s-1, 1600 rpm, room temperature, [MTBD][$N(SO_2CF_3)_2$]

- IL modified catalysts degrades only minor.
- Effects are: electrostatic stabilization; blocking of oxidation sites

Summary

Tuning carbon catalysts properties maters in thermal and electrocatalysis:

- pore size (distribution)
- surface chemistry
- graphitization
- metal dispersion
- smart additives

Acknowledgement

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