

# Porézní materiály pro adsorpci a katalýzu

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M U N I  
S C I



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# Mezoporézní metalosilikátové, organosilikátové a metalofosfátové materiály s vysokým měrným povrchem

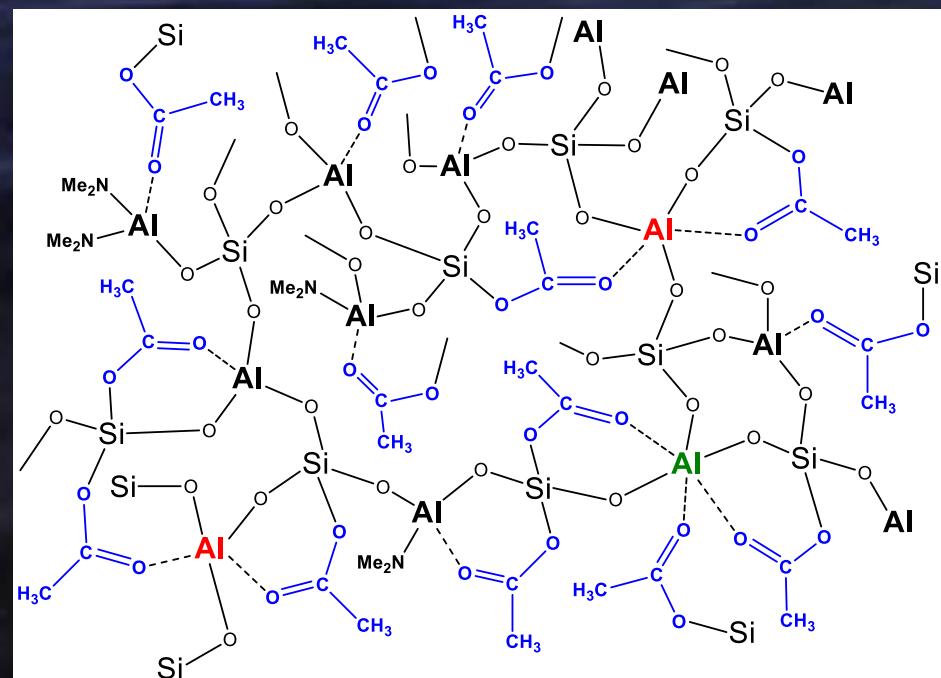
Sol-gelové metody - hydrolytické a nehydrolytické polykondenzační reakce

Hybridní organosilikáty

Fosforečnany křemičité

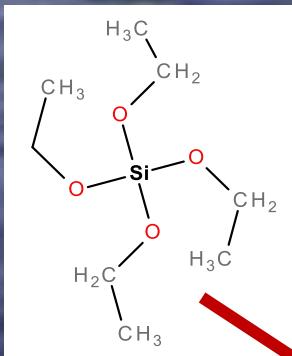
Metalokřemičitany

Hlinitofosforečnany a fosfonáty



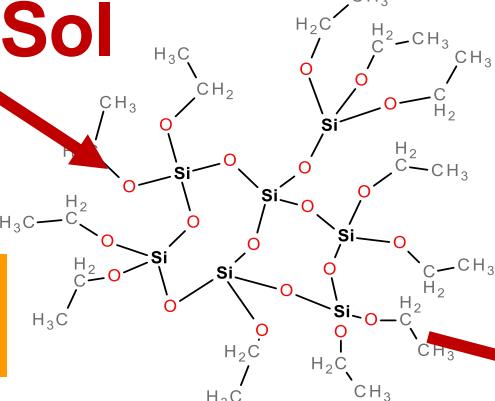
# Sol-Gelový proces

Roztok molekulárního prekurzoru



TEOS

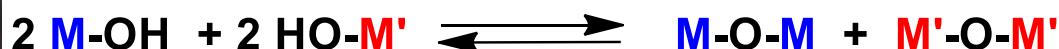
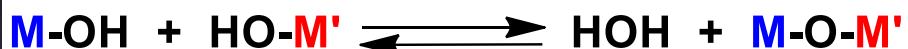
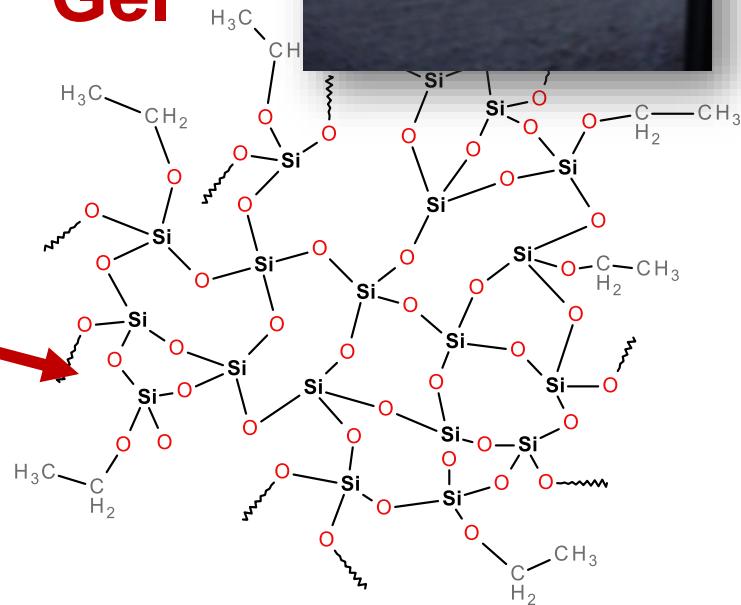
**Sol**



- 1) Hydrolýza
- 2) Polykondenzace

Koloidní roztok

**Gel**



Polymerní síť - silikagel

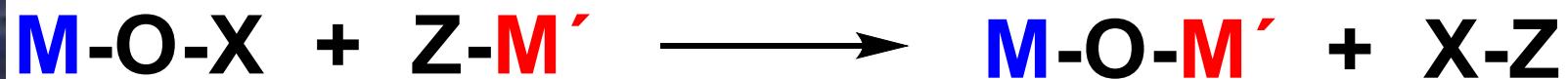
Mikro pod 2 nm  
Meso 2 - 50 nm



# Nehydrolytická sol-gelová metoda

Polykondenzace = eliminace malých molekul X-Z

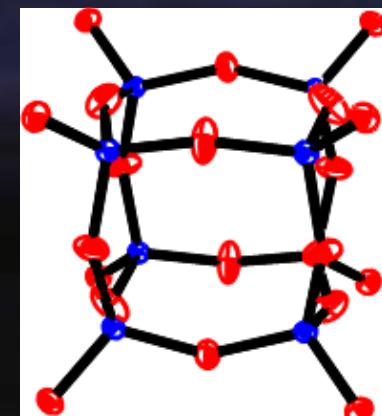
(alkyl halogenidy, ethery, estery, alkeny,...)



M, M' =

- monojaderné  
(P, Si, Al, Ti, Y, Zr, V, Nb, Mo, W, Fe, Zn, Sn,...)

- polyjaderné klastery  
 $\text{Ti}_8\text{O}_8(\text{O}_2\text{CR})_{16}$ ,  $\text{Si}_8\text{O}_{20}\text{R}_8$ ,  $\text{Al}_4\text{P}_4\text{O}_{12}\text{R}_8$



# Nové polykondenzační reakce

**Silicon acetates + metal amides**  $\text{Si}-\text{O}-\text{C}(\text{O})\text{CH}_3 + \text{R}_2\text{N}-\text{M} \rightarrow \text{Si}-\text{O}-\text{M} + \text{R}_2\text{N}-\text{C}(\text{O})\text{CH}_3$

**M = Al, Ti, Zr, Sn    Acetamide elimination**

**Silicon acetates + silyl esters**     $\text{Si}-\text{O}-\text{C}(\text{O})\text{CH}_3 + \text{Me}_3\text{Si}-\text{O}-\text{P} \rightarrow \text{Si}-\text{O}-\text{P} + \text{Me}_3\text{SiO}-\text{C}(\text{O})\text{CH}_3$

**Silylester elimination**

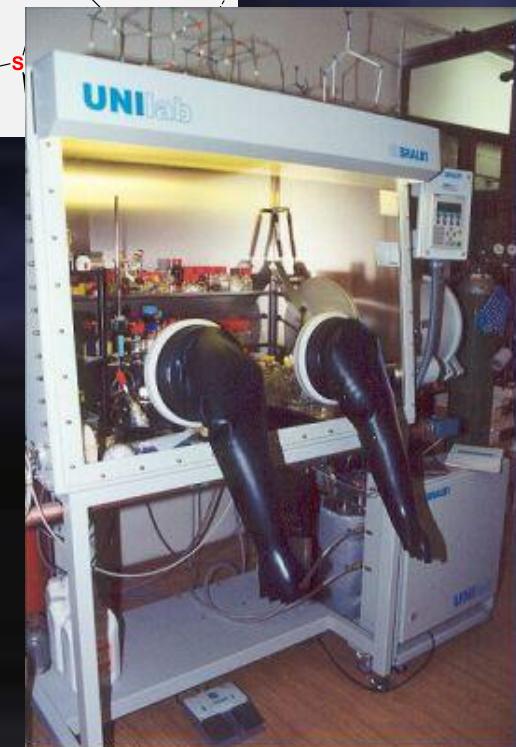
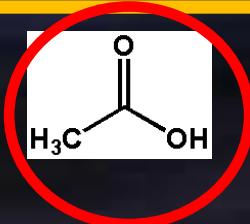
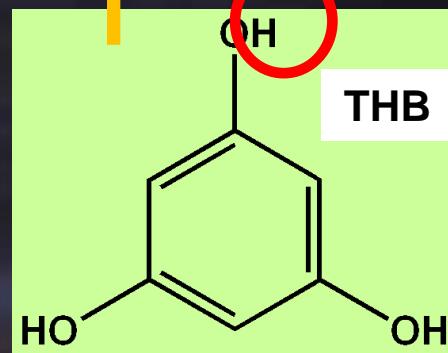
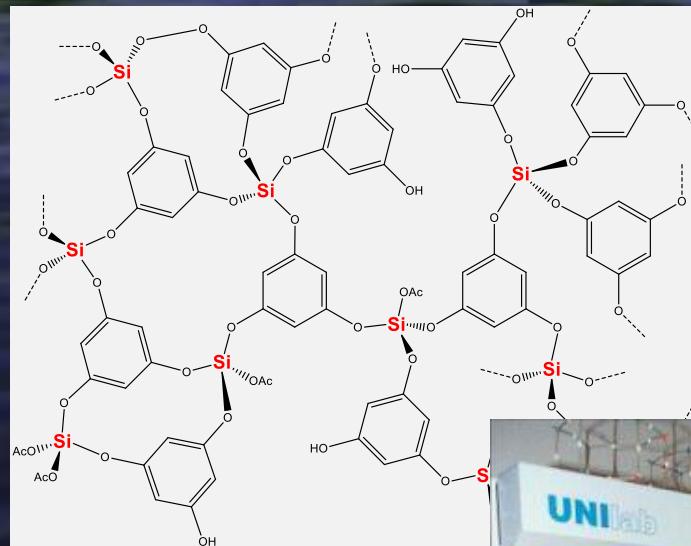
**Silicon acetates  
+ polyphenols**    **Si–O–C(O)CH<sub>3</sub>** + HO–C → **Si–O–C** + **HO–C(O)CH<sub>3</sub>**

**Silyl esters**  
**+ metal amides**  $P-O-SiMe_3 + R_2N-Al \rightarrow P-O-Al + R_2N-SiMe_3$   
**Silylamine elimination**

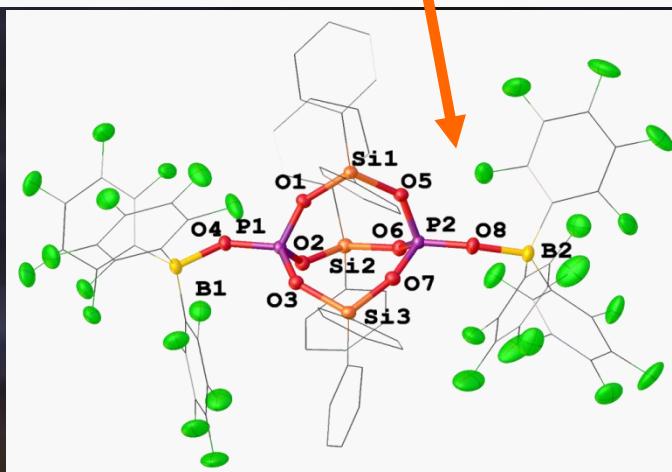
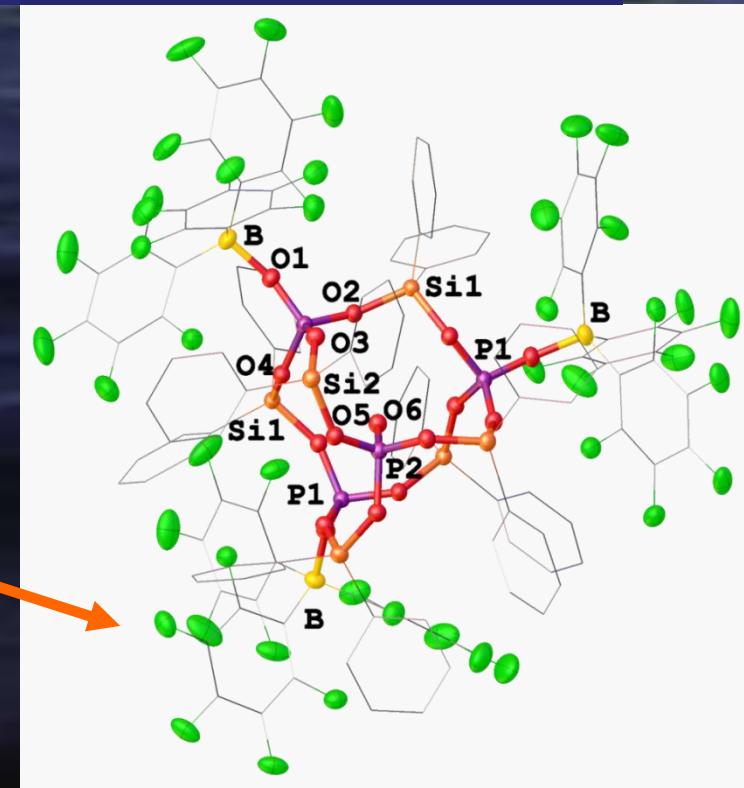
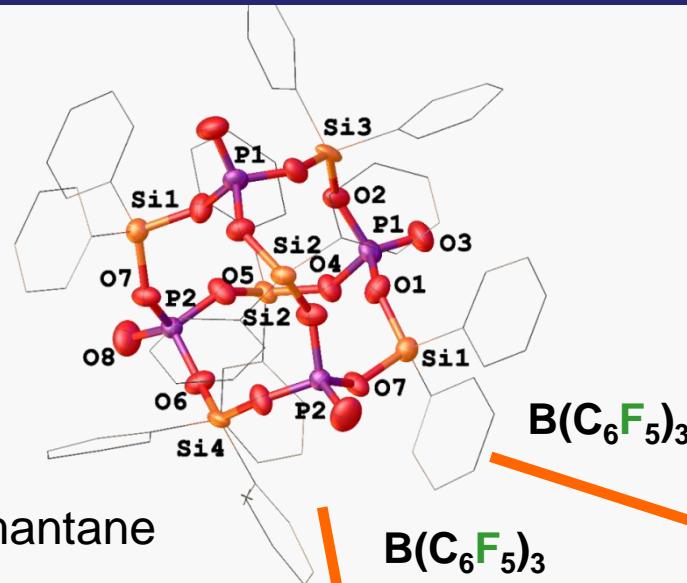
# Polykondenzace s eliminací HOAc



## 1,4-dioxan, 100 °C, 7 dní



# Molekulární Organosilikofosfáty

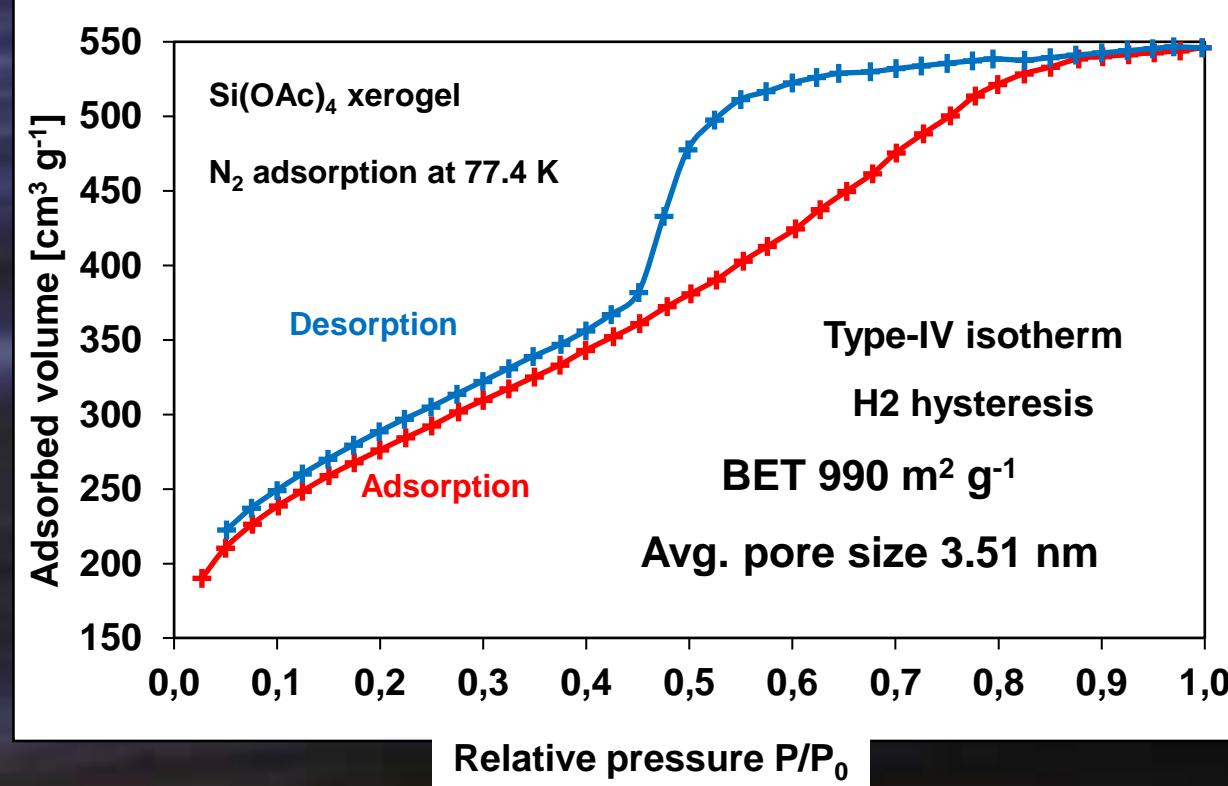


A. Styskalik, M. Babiak, P. Machac, B. Relichova,  
J. Pinkas\* *Inorg. Chem.* 2017, 56, 10699–10705

# Charakterizace

Adsorpce-desorpce dusíku, argonu, oxidu uhličitého a vodní páry

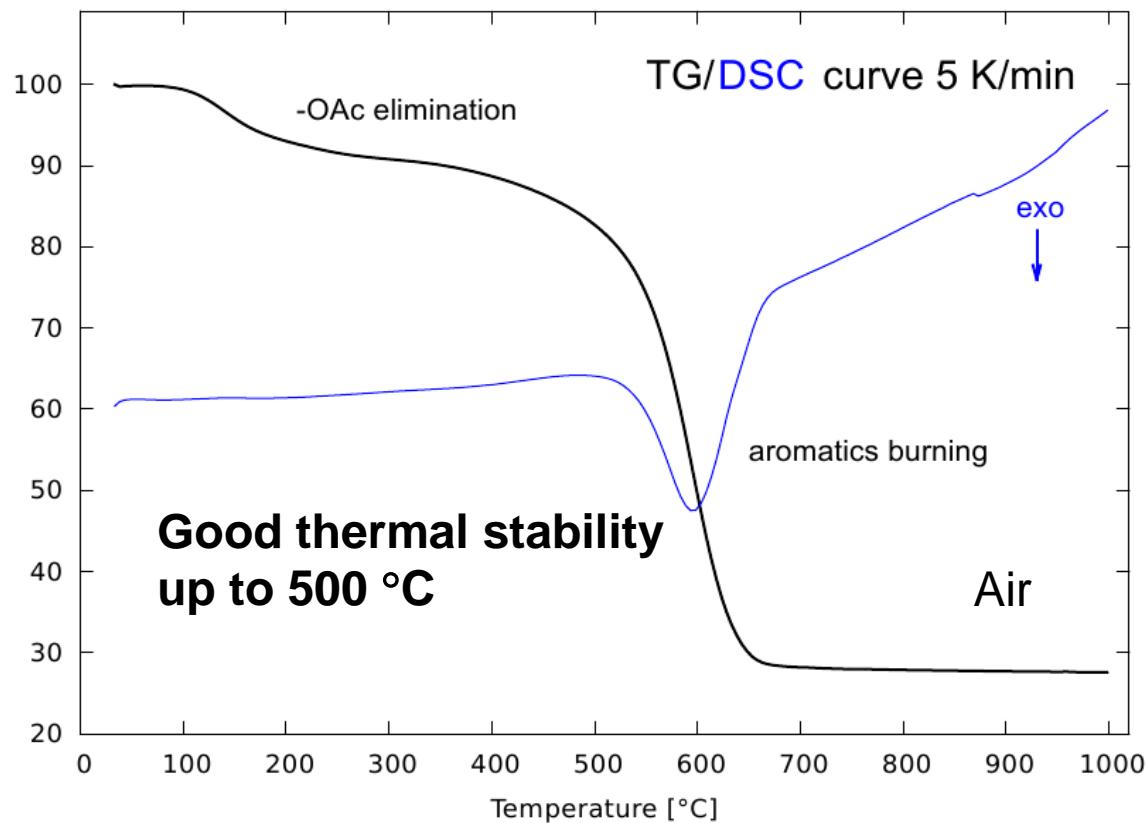
- Porozita - velikost pórů
- Měrný povrch
- Tvar pórů
- Katalytické vlastnosti



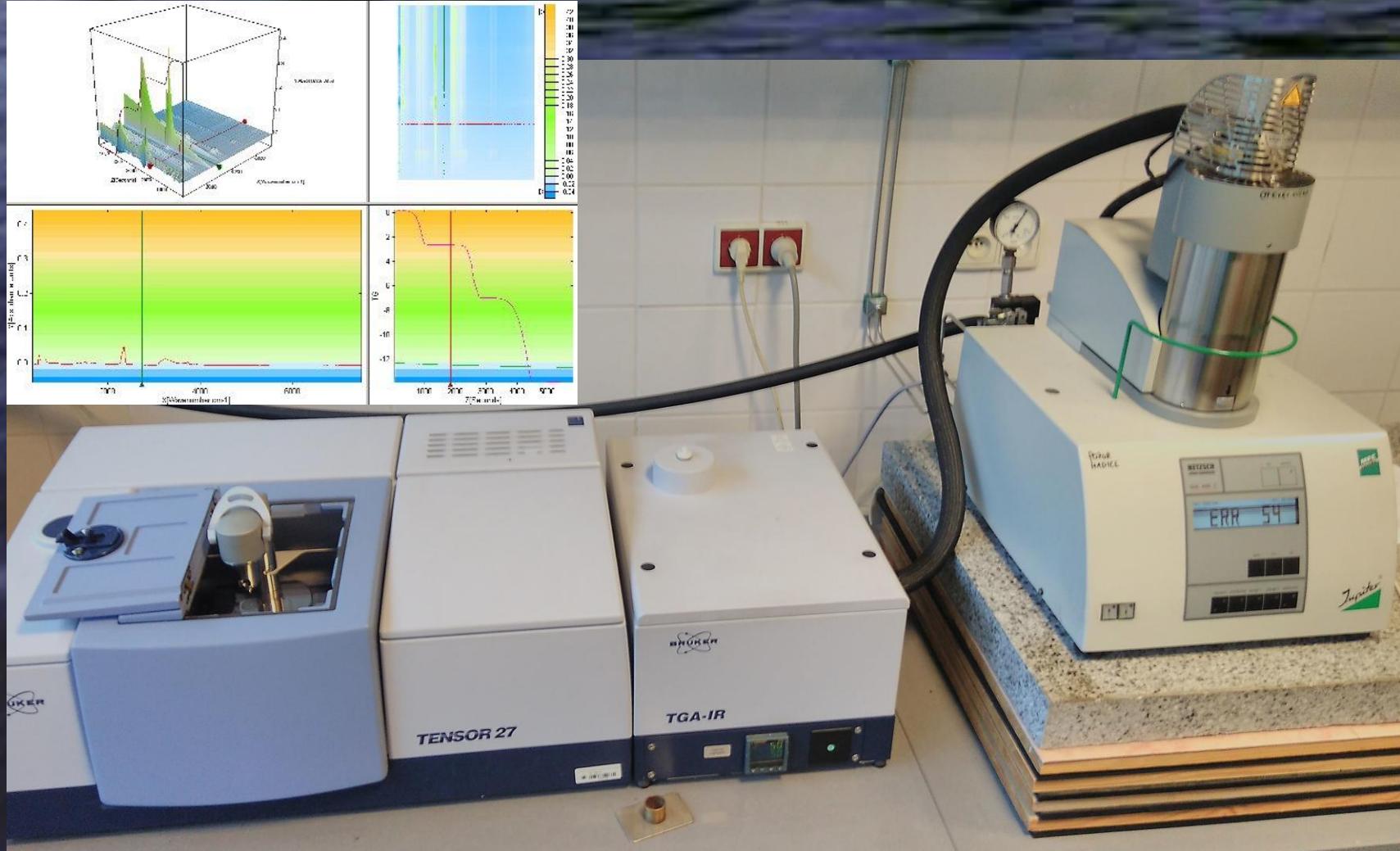
# Charakterizace

## Termogravimetrie a diferenční skenovací kalorimetrie

- Termická stabilita
- Krystalizace
- Unikající plyny



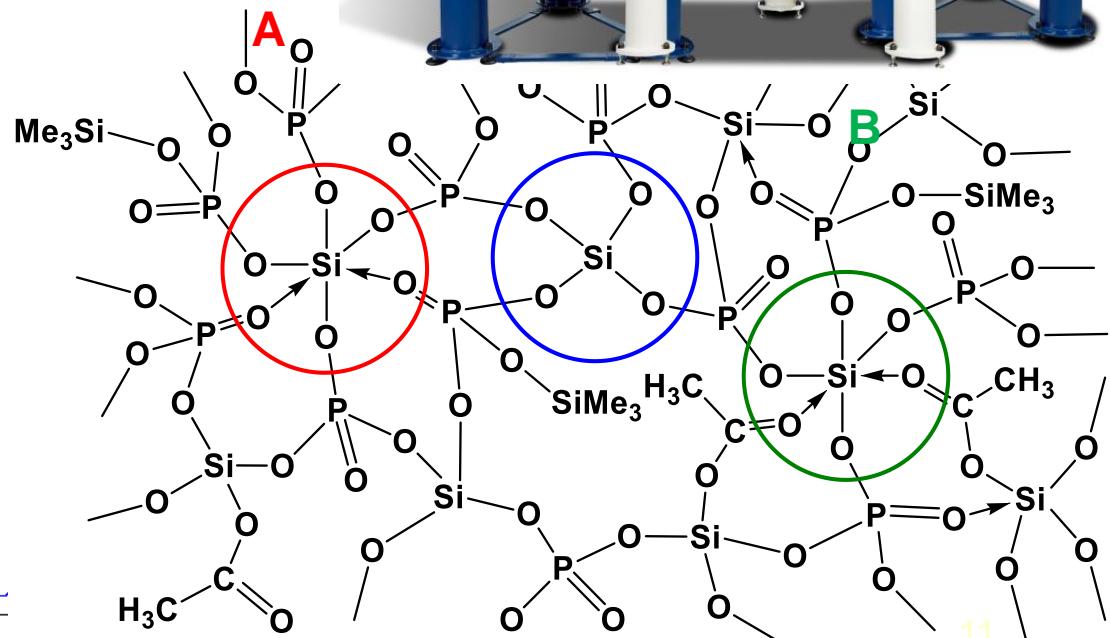
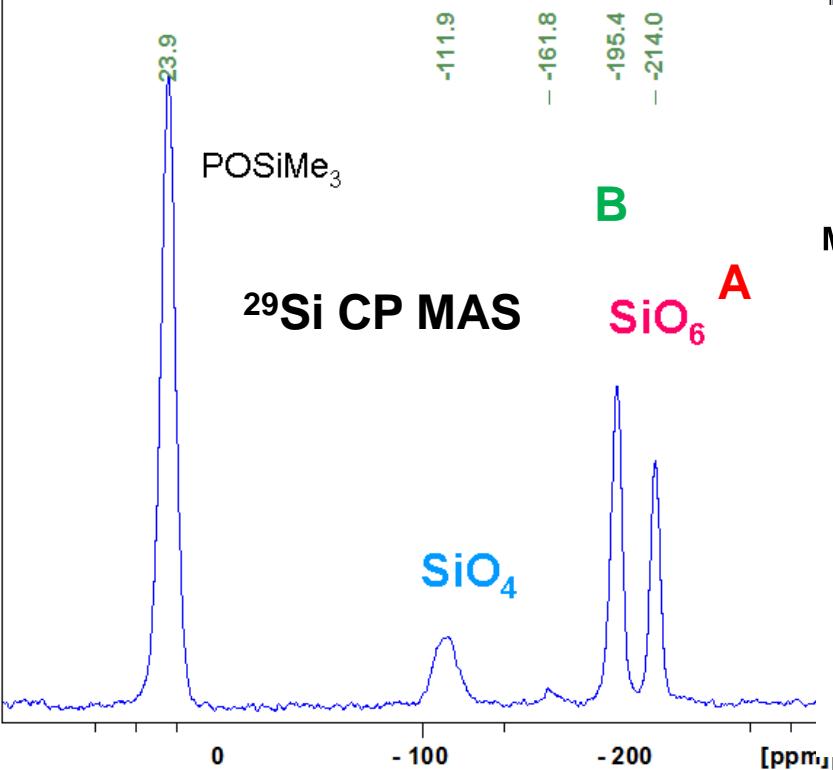
# Charakterizace



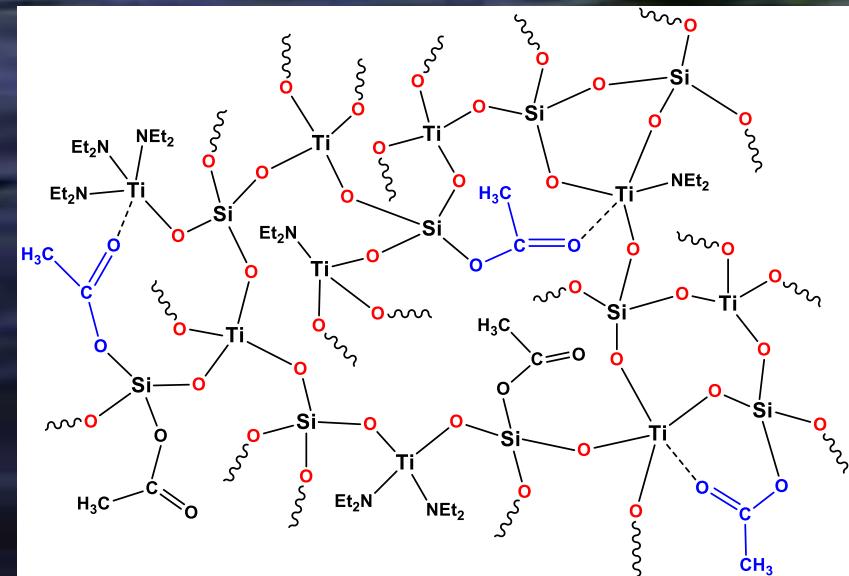
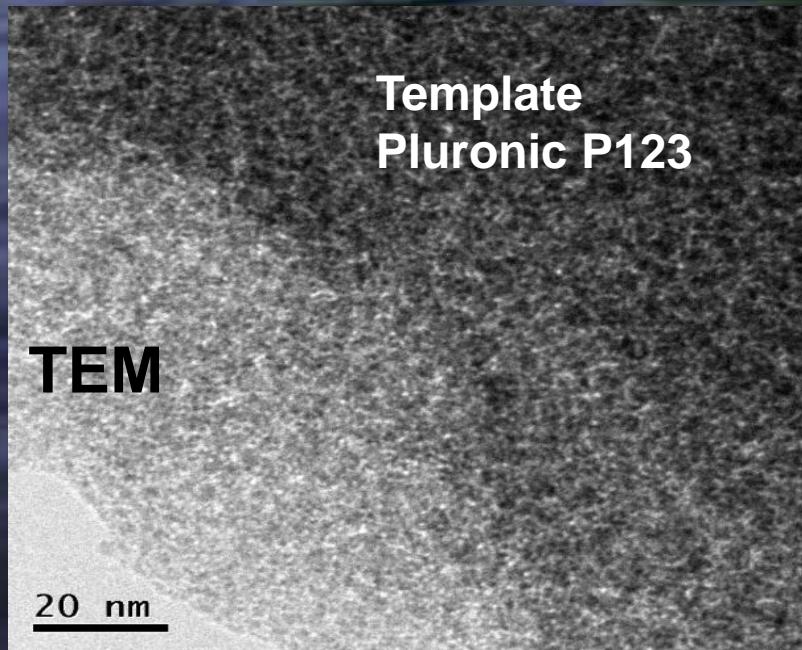
# Charakterizace

## NMR spektroskopie v pevné fázi

- Chemicky odlišné atomy
- Koordinační číslo



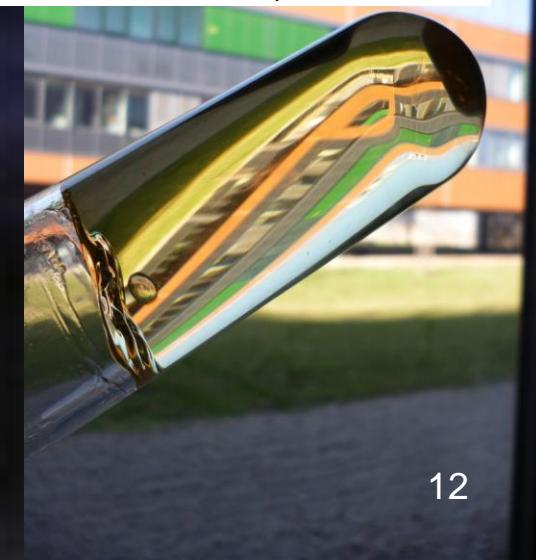
# Charakterizace



**Wormhole structure**  
Surface areas:  $326\text{--}615 \text{ m}^2 \text{ g}^{-1}$   
Pore diam: 2.6–7.4 nm

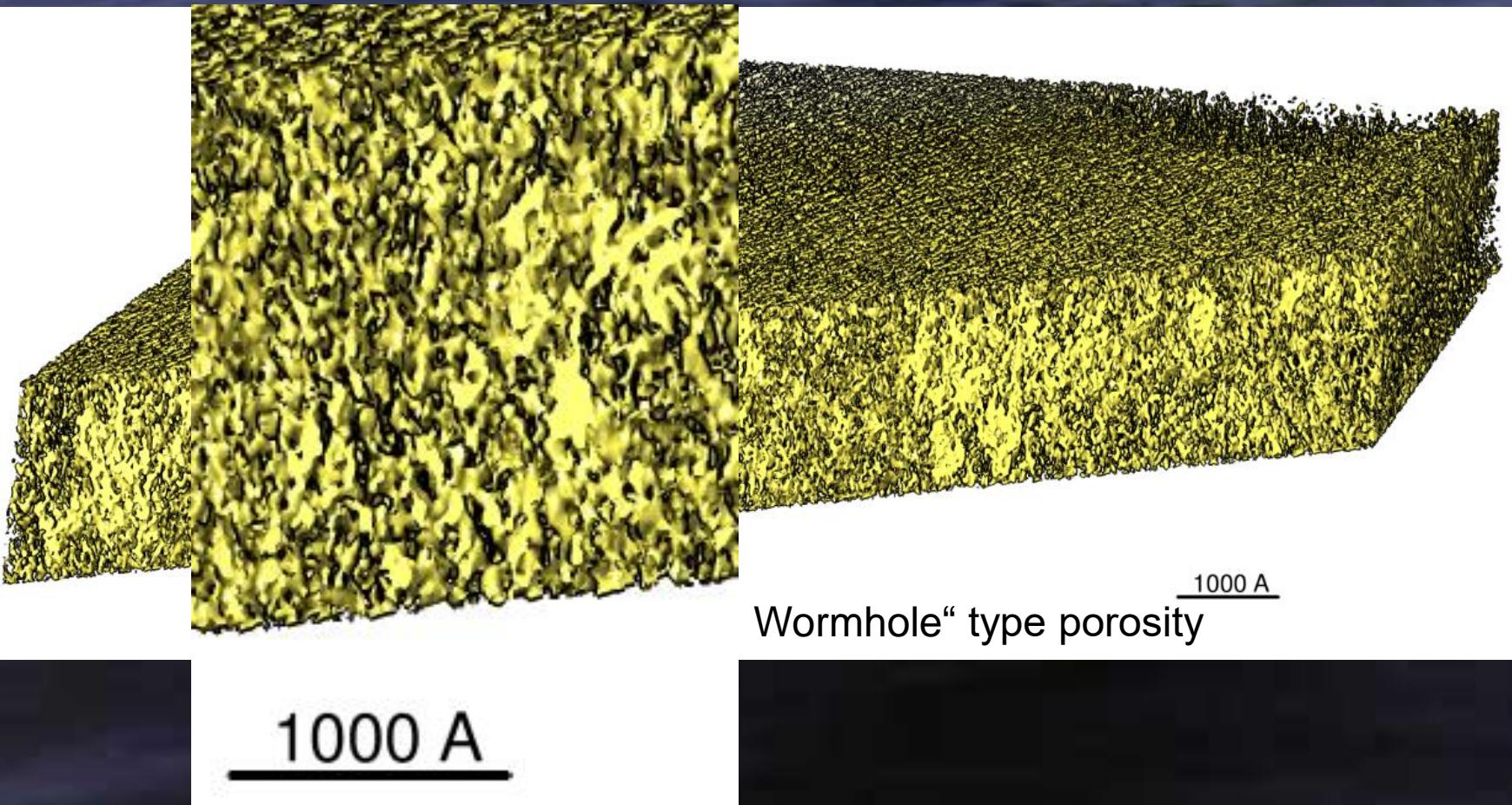
TEM - transmисní elektronová mikroskopie

- Velikost pórů
- Tvar částic a pórů



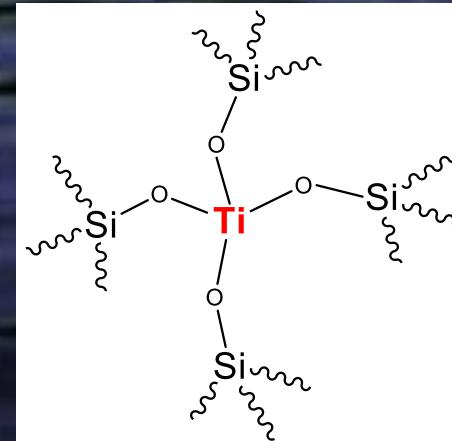
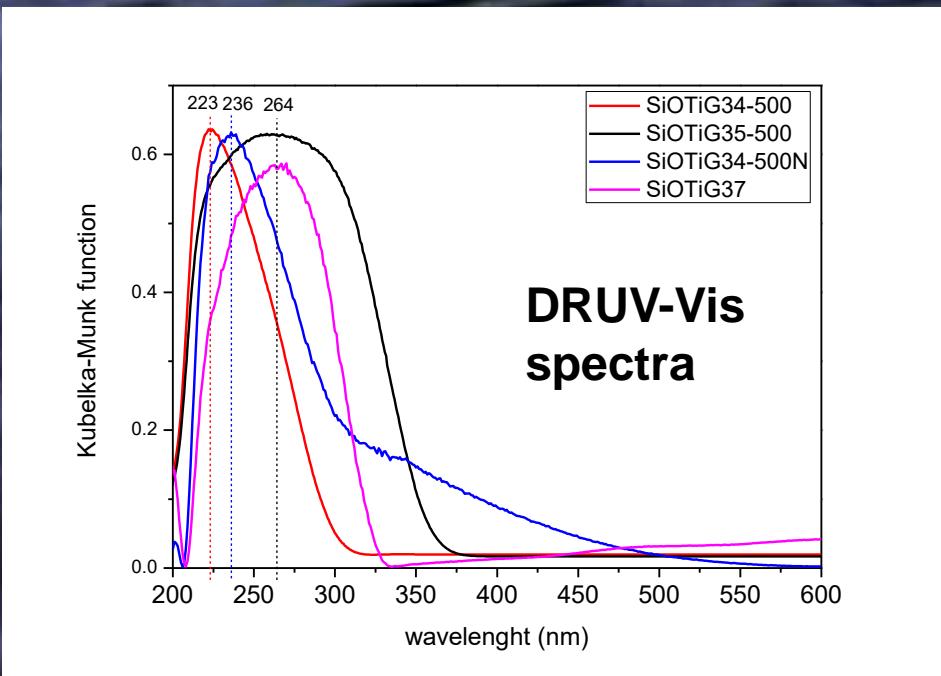
# TEM Tomografie

3D model reconstruction

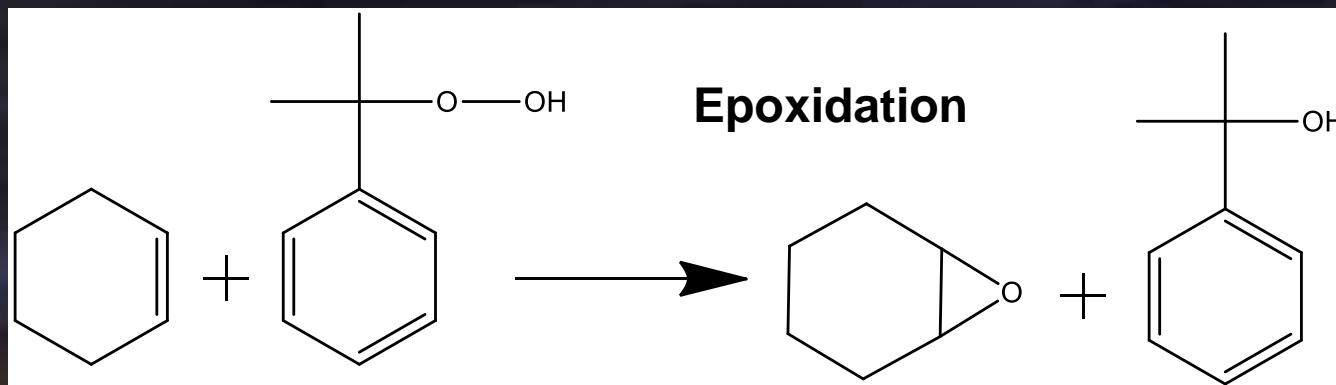


3D rendering of a reconstructed tomogram of zirconosilicate xerogel

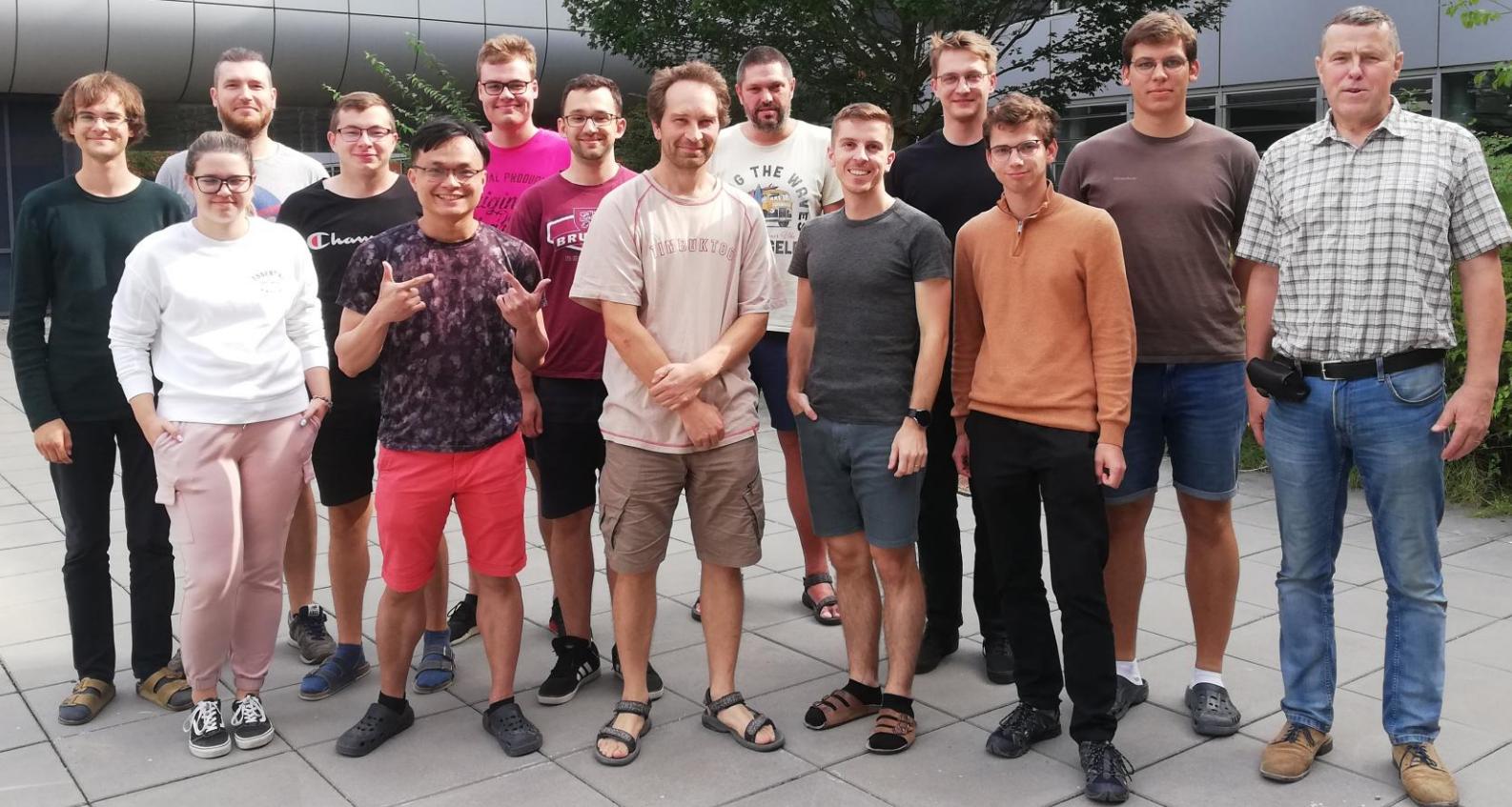
# Titanosilikáty jako katalyzátory



**The best catalyst**  
templated, calcined, ~10 % Ti  
conversion 96 %,  
>99 % selectivity (**no alcohols**)



Děkuji za pozornost



Kontakt: [jpinkas@chemi.muni.cz](mailto:jpinkas@chemi.muni.cz)