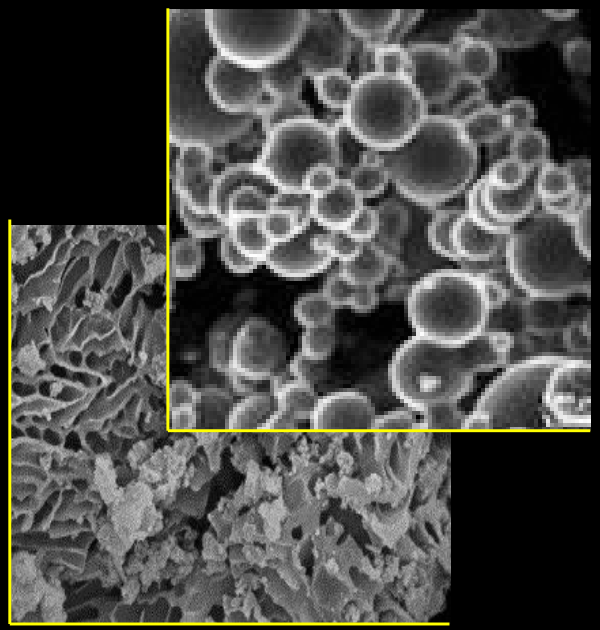
Porózus anyagok : aerogélek - kriogélek



ELTE
Kémiai Intézet
Sinkó Katalin

Gélek előállítása

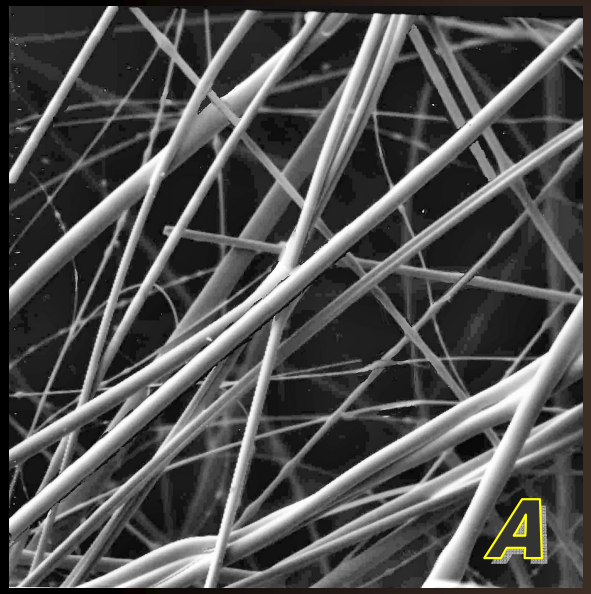
$Al(NO_3)_3 \cdot 9H_2O$ + 1-propanol

Optimális összetétel:

Al-nitrát / propanol mólaránya = 1 : 4,5

propanol / víz mólarány = 1 : 2

Al(III)-koncentráció = ≈ 2 mól dm^{-3}



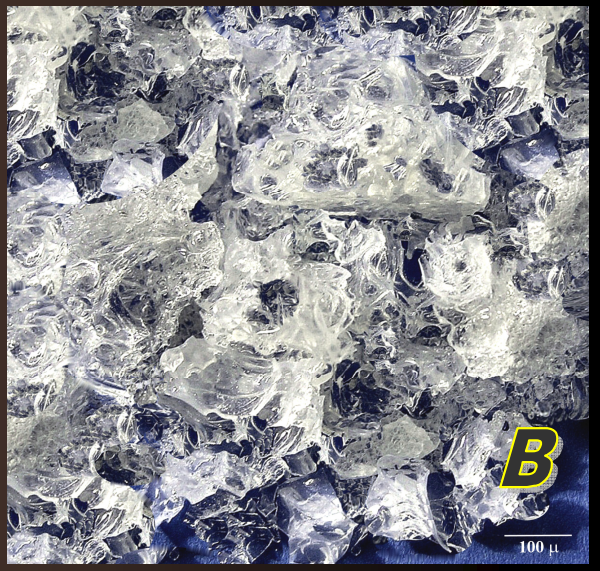
szálasítható, viszkózus A

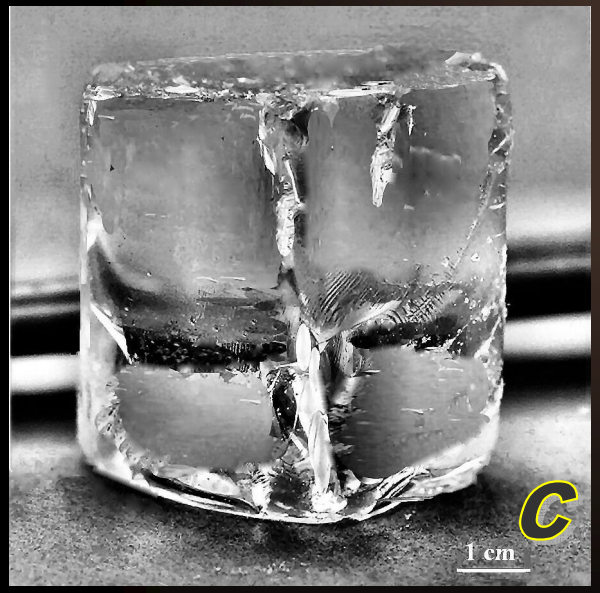
szárítás ↓ *80°C (∑72 m/m%)*

makropórusos hab B

duzzasztás ↓ *víz (0-100 m/m%)*

optikailag tiszta hidrogél C





Gélek előállítása

$Al(NO_3)_3 \cdot 9H_2O$ + 1-propanol

reflux — **24 h**, *80 °C*

bepárlás — *60 °C (∑60 m/m%)*

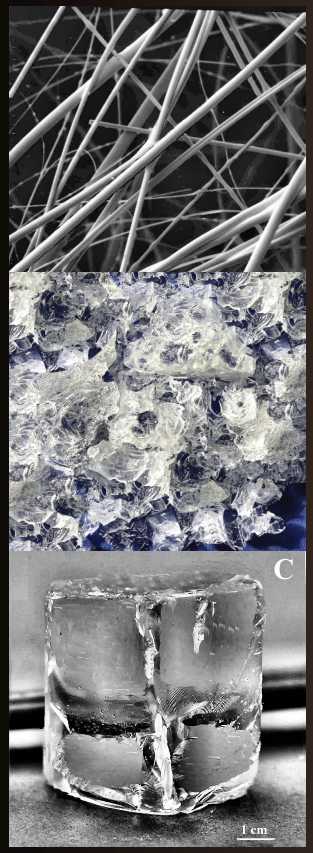
szálasítható, viszkózus A — nitrát/Al^{3+} = 1.2 mol, $[Al^{3+}]$ = 9.5 m/m% — **A**

szárítás — *80 °C (∑72 m/m%)*

makropórusos hab B — nitrát/Al^{3+} = 0.85 mol, $[Al^{3+}]$ = 14 m/m% — **B**

duzzasztás — *víz (0-100 m/m%)*

optikailag tiszta hidrogél C — $[Al^{3+}]$ = 1-14 m/m% — **C**



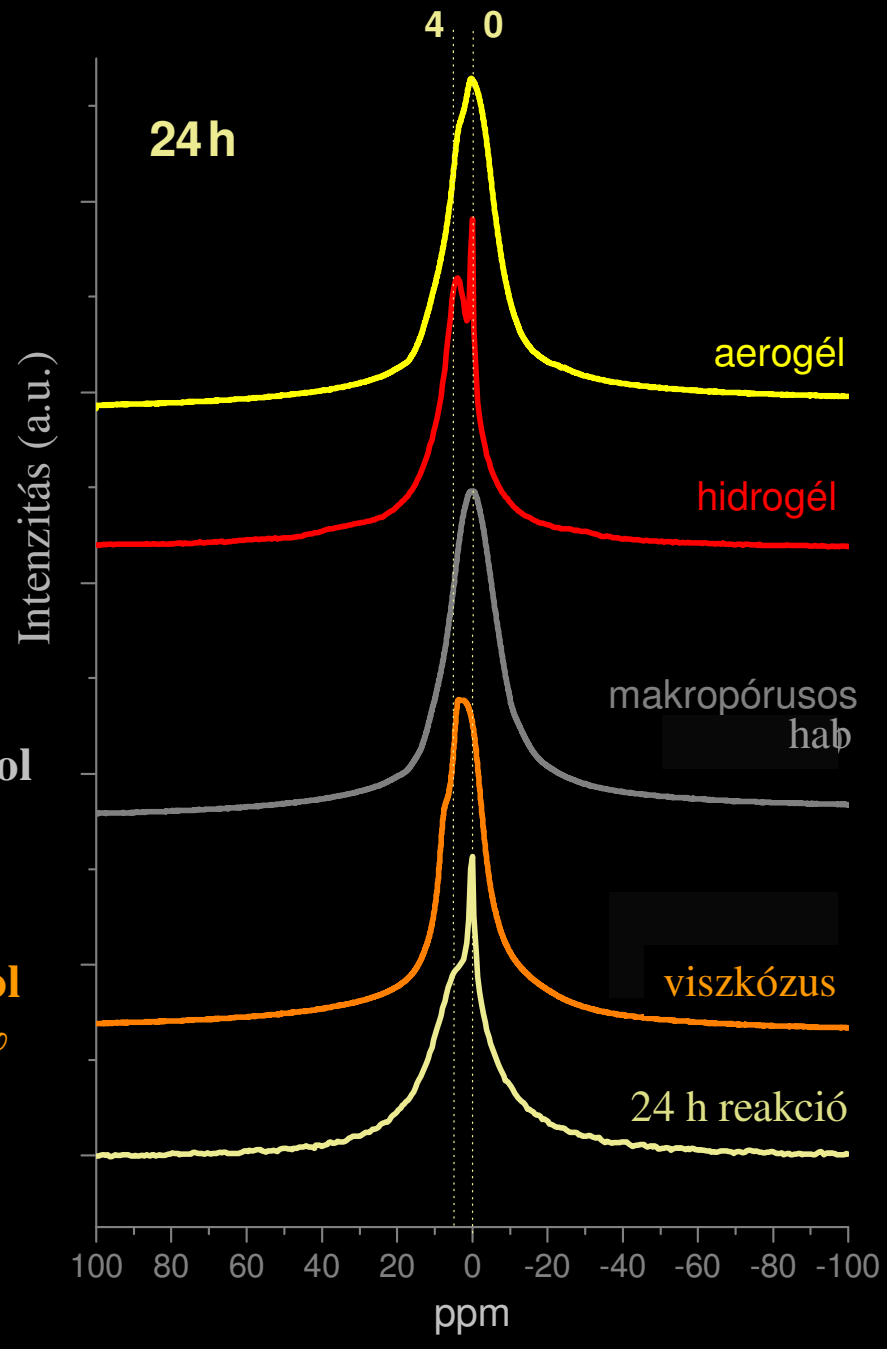
szuperkritikus szárítás — *313 K, 10 MPa*

fagyasztva szárítás, $N_{2(liq)}$

nitrát/Al^{3+} = 0.85 mol, $[Al^{3+}]$ = 14 m/m% — **aerogél D**

kriogél E — nitrát/Al^{3+} = 0.85 mol, $[Al^{3+}]$ = 10-12 %

Gélek szerkezete - Al MAS NMR



nitrát/Al^{3+} =0.85 mol
[Al^{3+}] = 14 m/m%

[Al^{3+}] = 1-14 m/m%

nitrát/Al^{3+} = 0.85 mol
[Al^{3+}] = 14 m/m%

nitrát/Al^{3+} = 1.2 mol
[Al^{3+}] = 9.5 m/m%

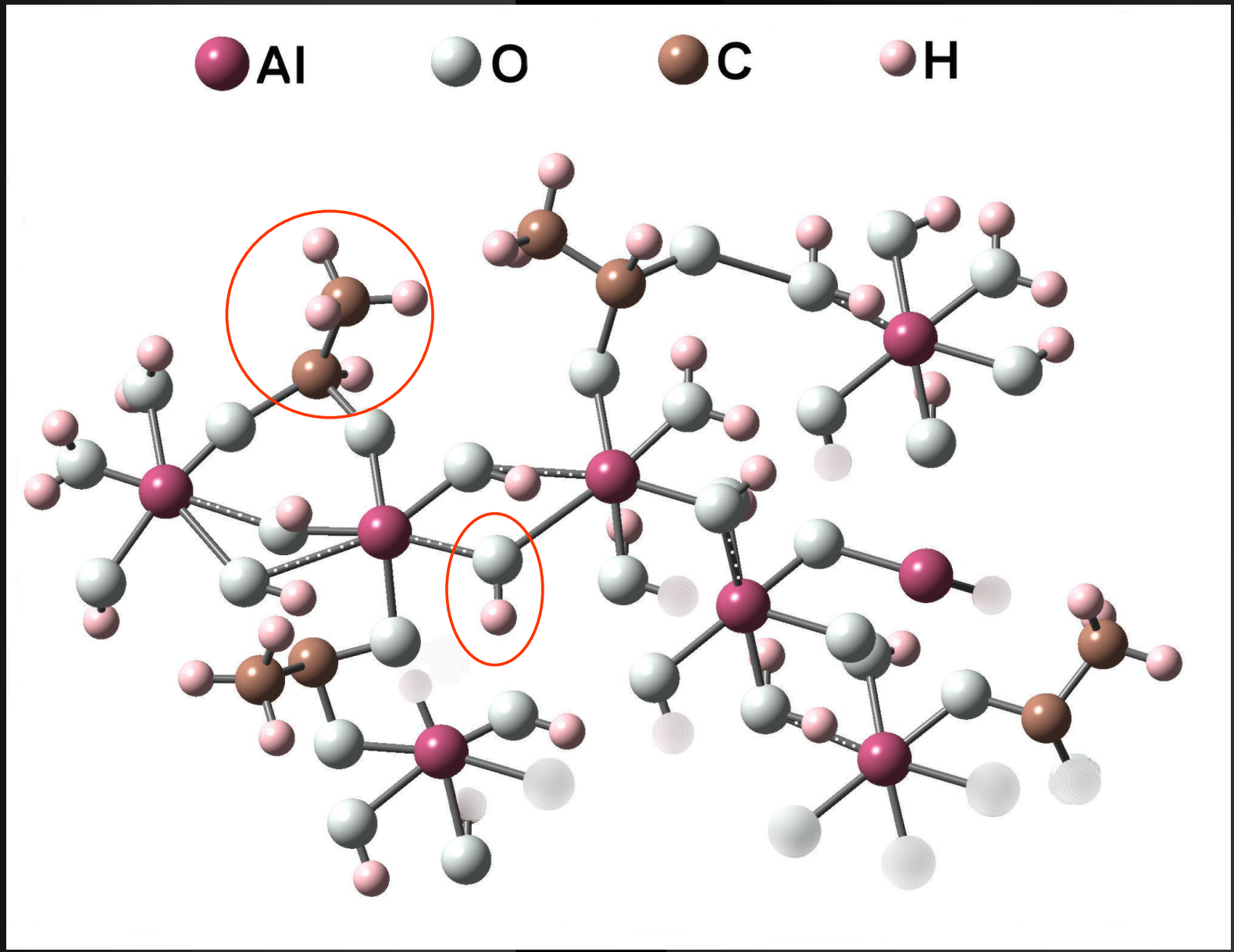
D
-3 ppm: 15%
3-4 ppm: 35%
8,5 ppm: 50%

C
0 ppm: 5-15%
4 ppm: 85-95%

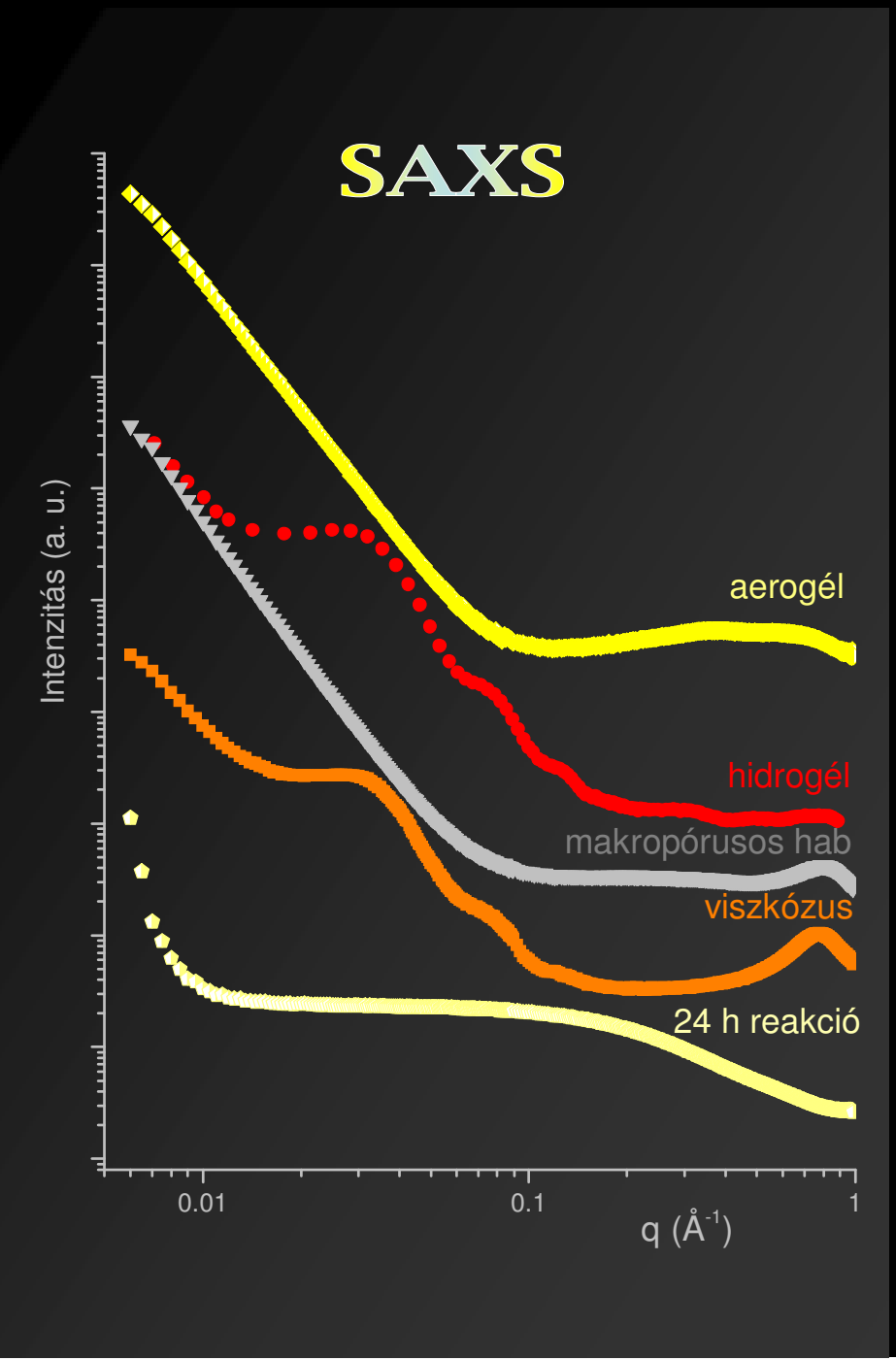
B
0 ppm: 100%

A
-3 ppm: 83%
3-4 ppm: 10%
8,5 ppm: 7%

AlOOH gélek kötésszerkezete - NMR FTIR



AlOOH gélek szerkezete - SAXS - WAXS

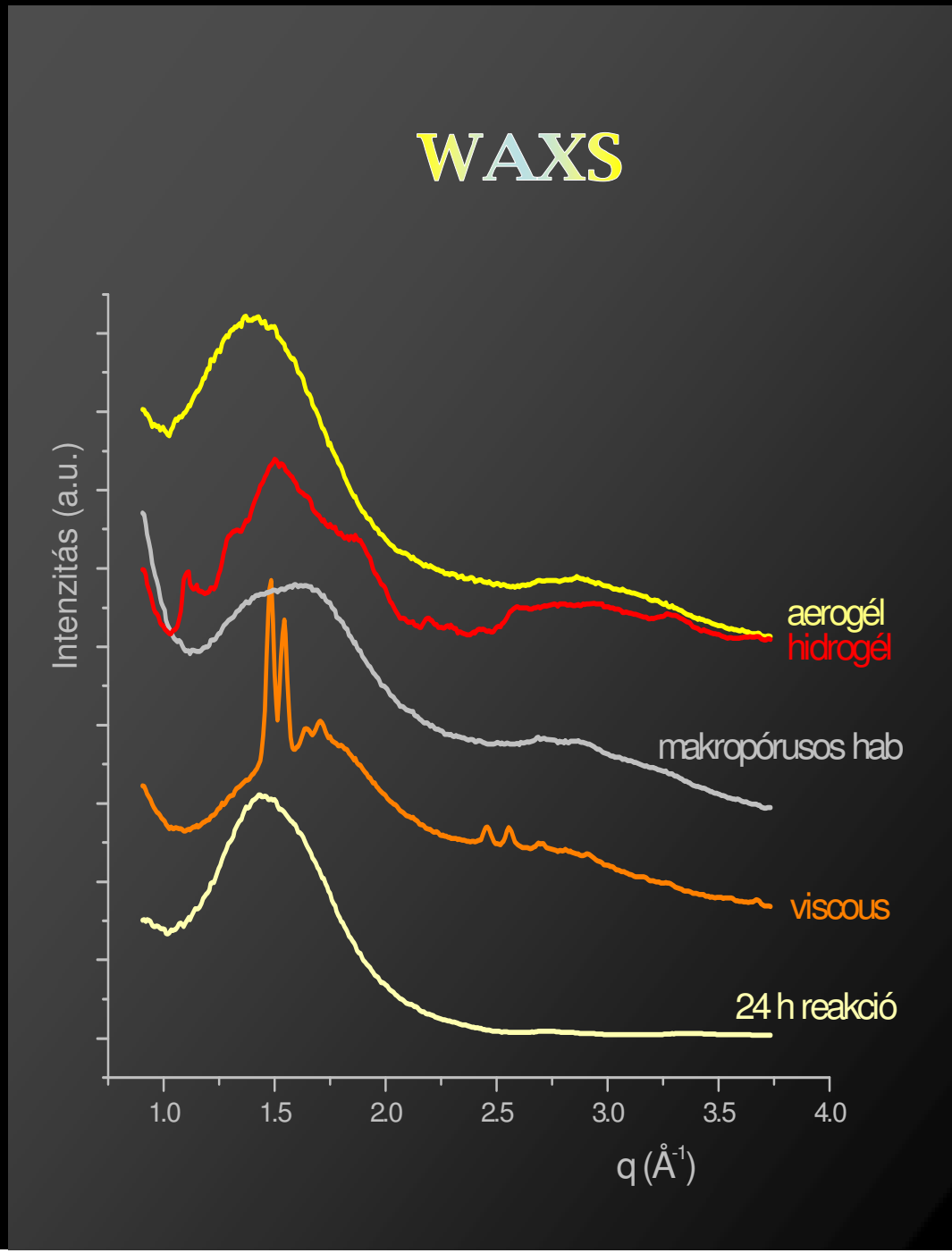


D

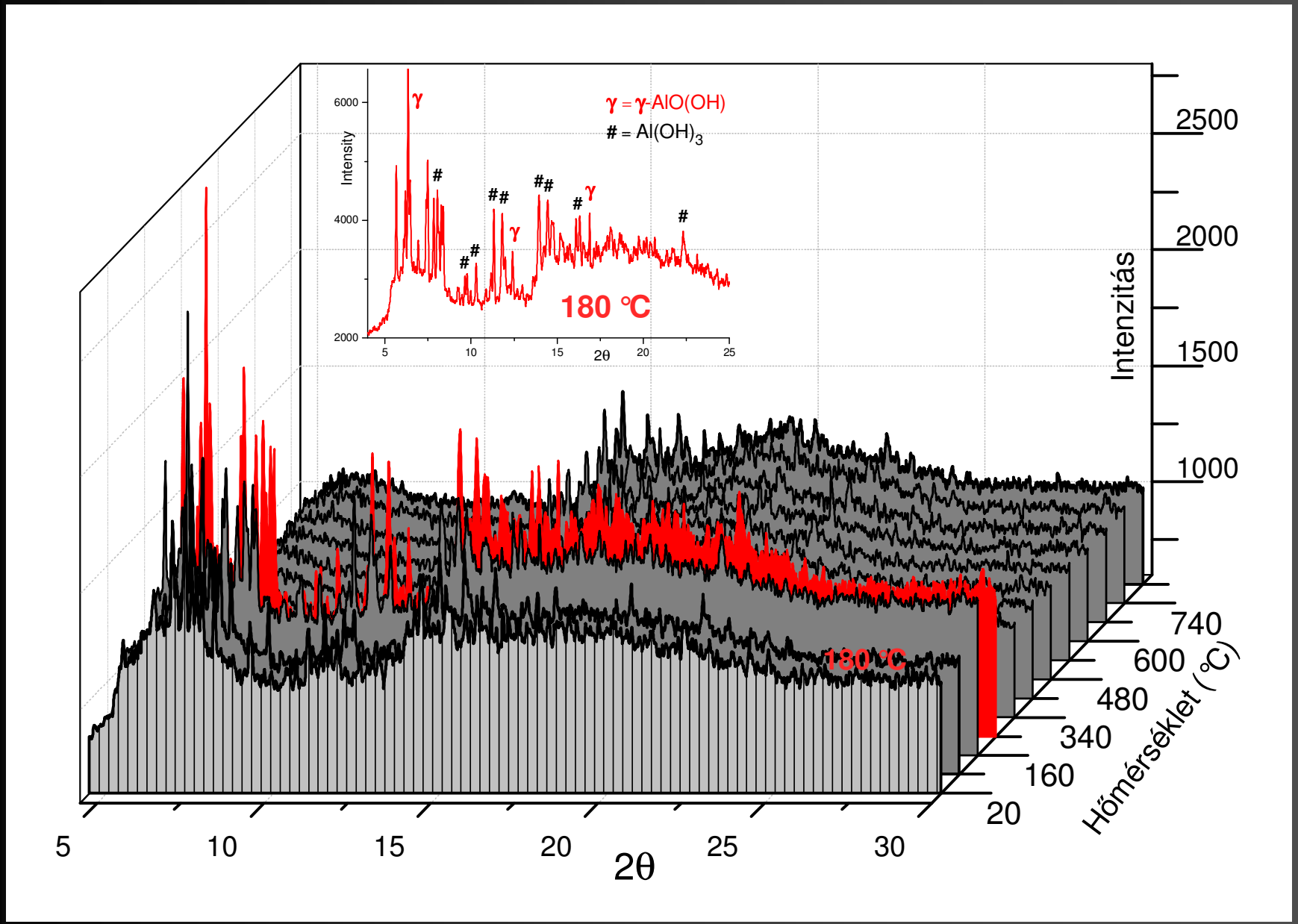
C

B

A

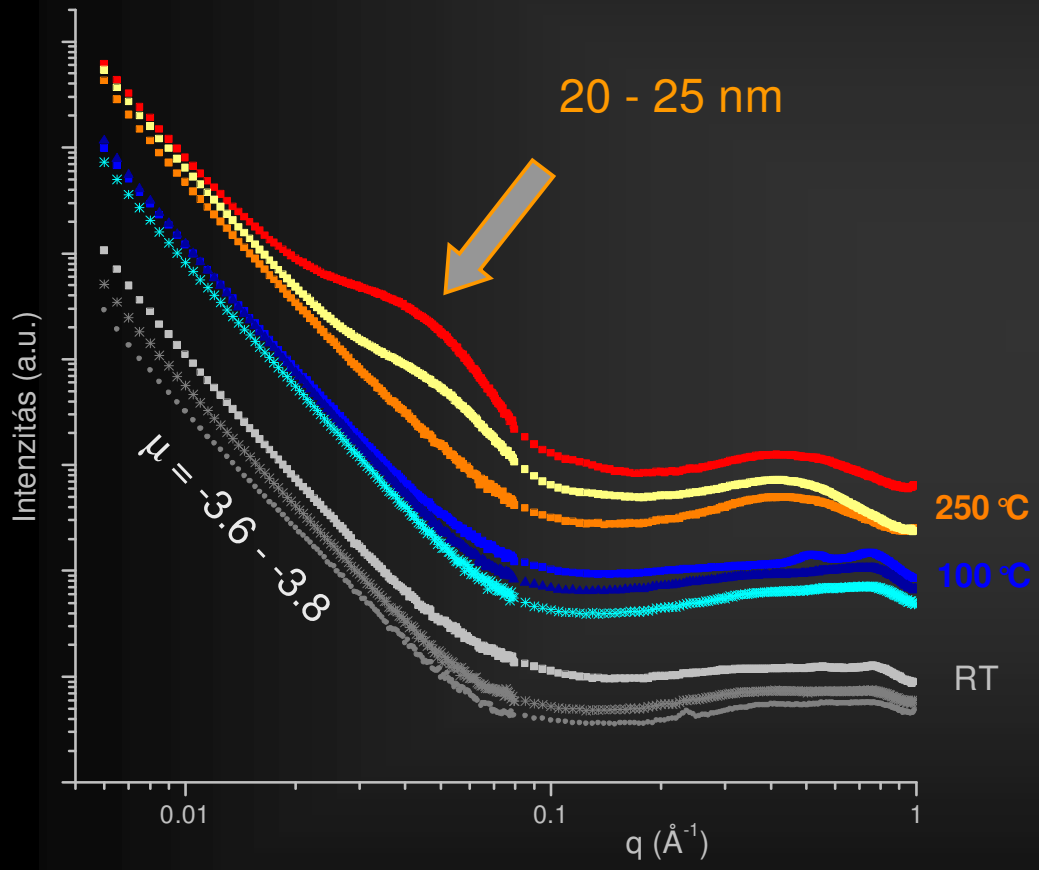


Gélek szerkezete - XRD

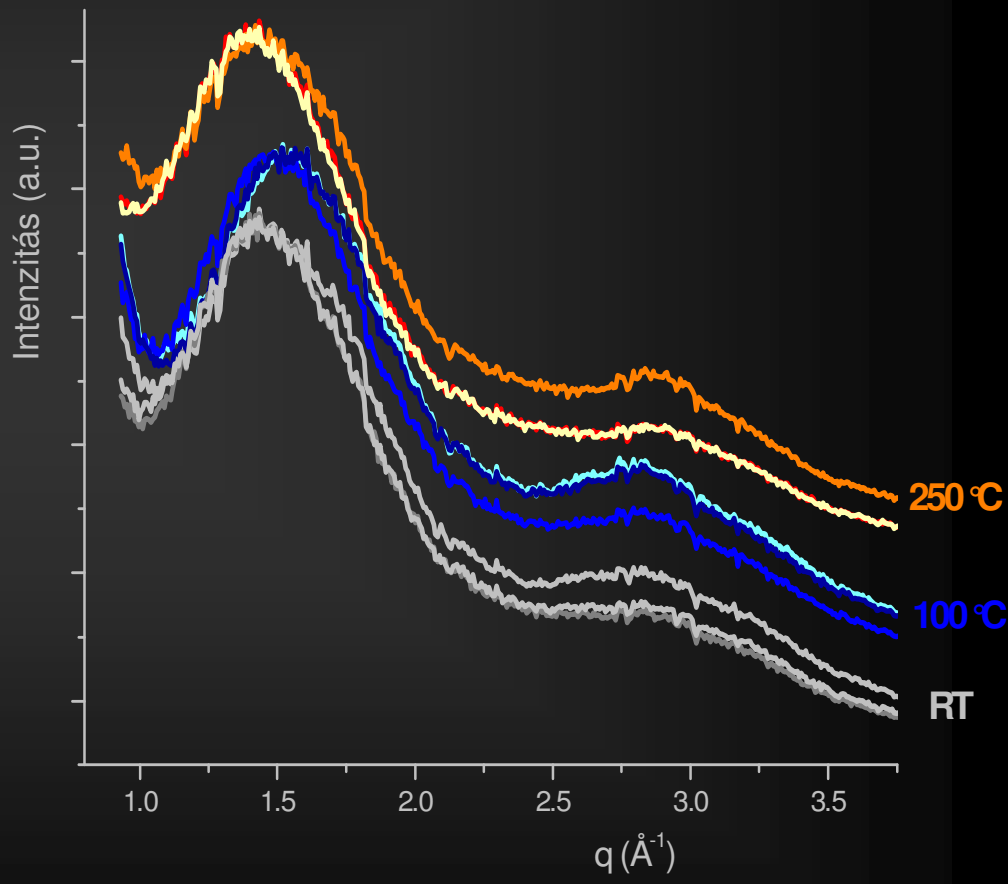


Kriogélek szerkezete - SAXS - WAXS

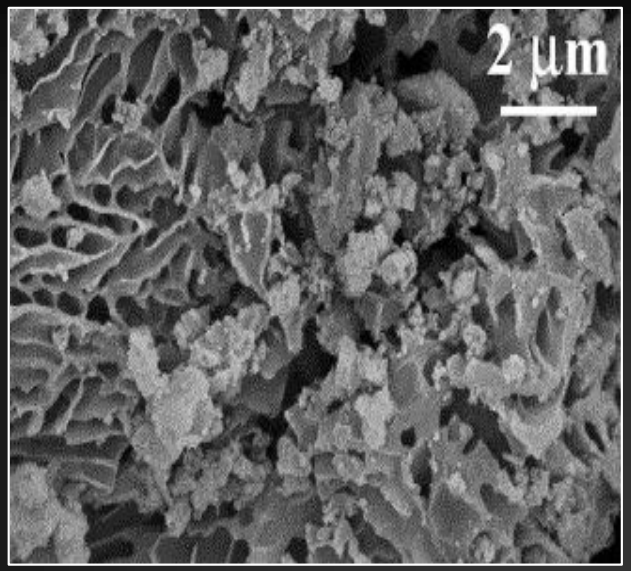
SAXS



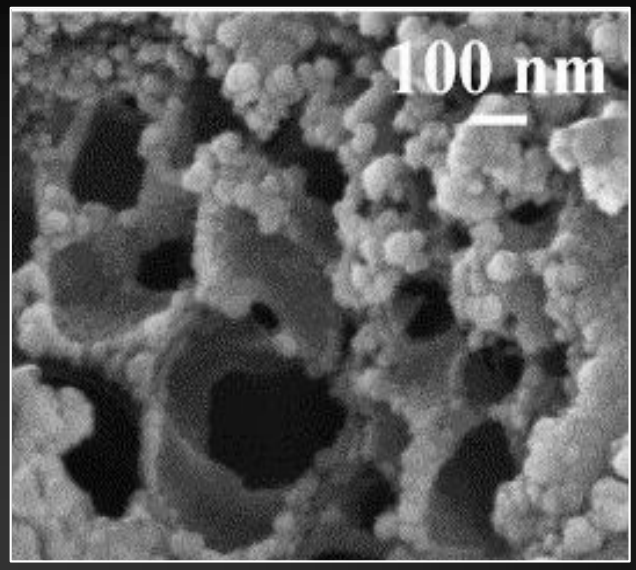
WAXS



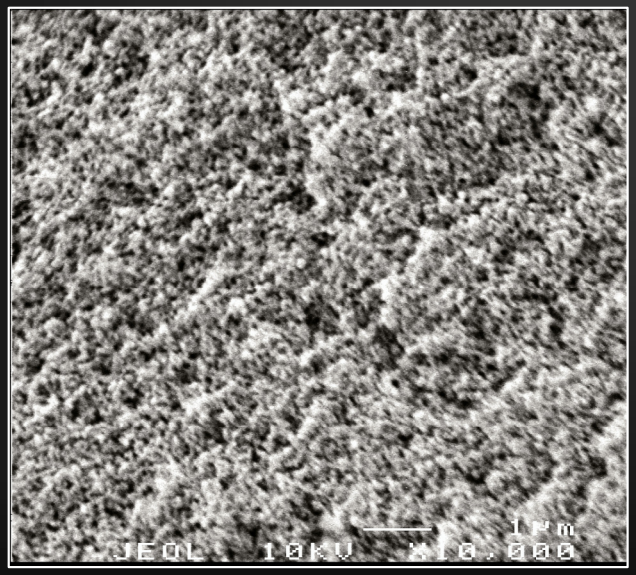
Porózus gélszerkezetek - SEM



kriogélek



20



10 000x-es felület

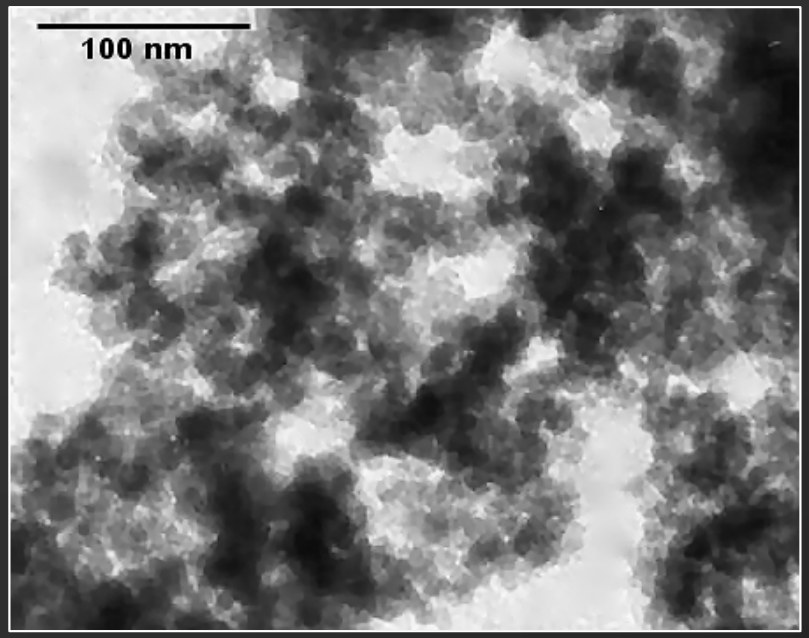
aerogélek

10 000x-es felület

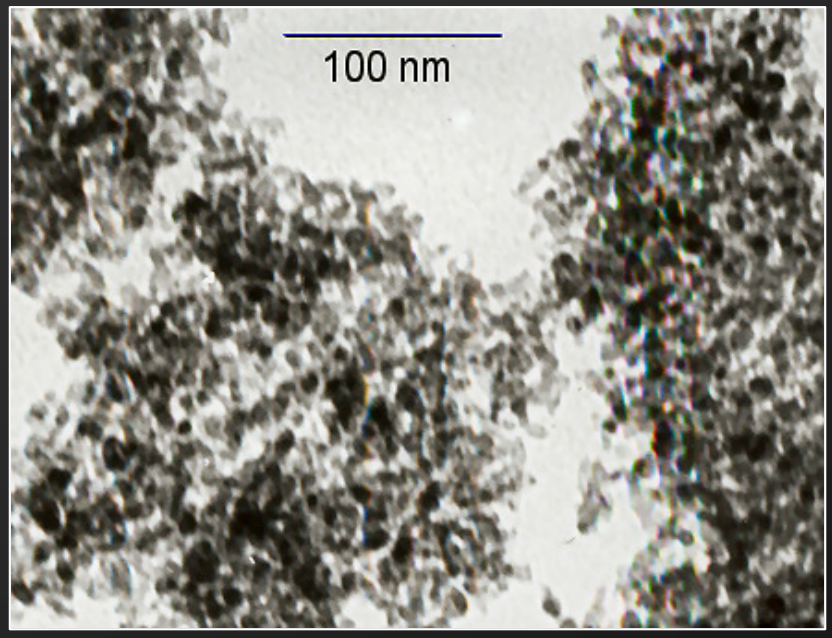
6

Aerogélek szerkezete - TEM

alumínium-szilikát



kxlaszterméret, ∅: 15 – 20 nm



szilika

részecskesugár: 5 – 6 nm (TEM)
5.6 nm (Guinier)

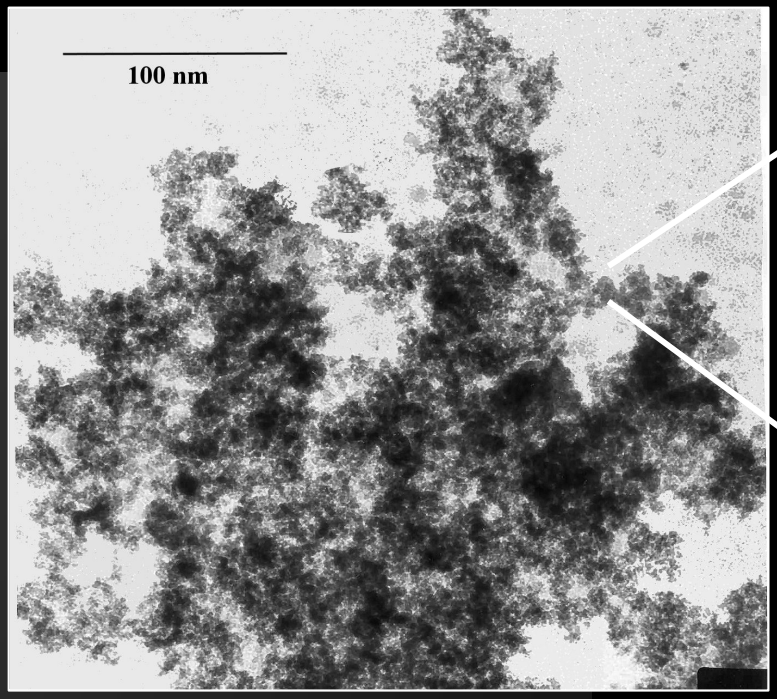
Aerogélek SAXS-felvételeinek kiértékelése **Freltoft**-formulával

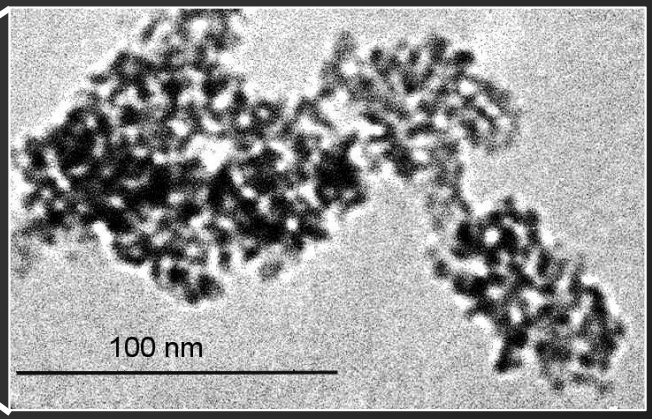
$$I(q) = back + A\left(1+\frac{\sqrt{2}}{3}q^2R^2\right)^{-2}\left(1+\frac{C\cdot\xi^D}{q\xi}\left(1+q^2\xi^2\right)^{\frac{1-D}{2}}\cdot\Gamma(D-1)\sin\left[(D-1)\operatorname{arctg}(q\xi)\right]\right)$$

korrelációs hossz (ξ) = 9 nm

elemi részecske (R) = 1.5 nm

Aerogélek szerkezete - TEM





elemi részecskeméret, ∅ = 6.0 nm
klaszterméret, ∅ = 50 – 60 nm (TEM)

$$I(q) = back + A\left(1+\frac{\sqrt{2}}{3}q^2R^2\right)^{-2}\left(1+\frac{C\cdot\xi^D}{q\xi}\left(1+q^2\xi^2\right)^{\frac{1-D}{2}}\cdot\Gamma(D-1)\sin\left[(D-1)\operatorname{arctg}(q\xi)\right]\right)$$

$$P(q) = \frac{1}{\left(1+\frac{\sqrt{2}}{3}q^2{r_F}^2\right)^2}$$

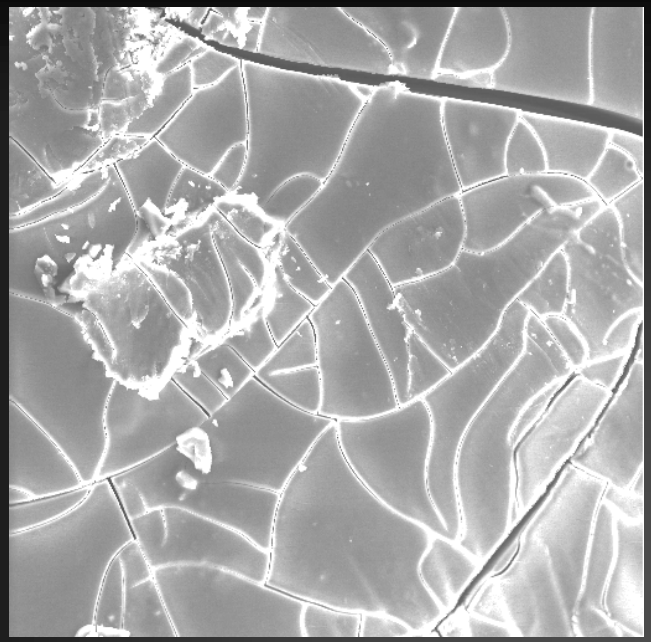
$$P(q) = \frac{1}{\left(1+\frac{\sqrt{2}}{3}q^2{r_F}^2\right)^{2-\alpha}}$$

módosított form-faktor

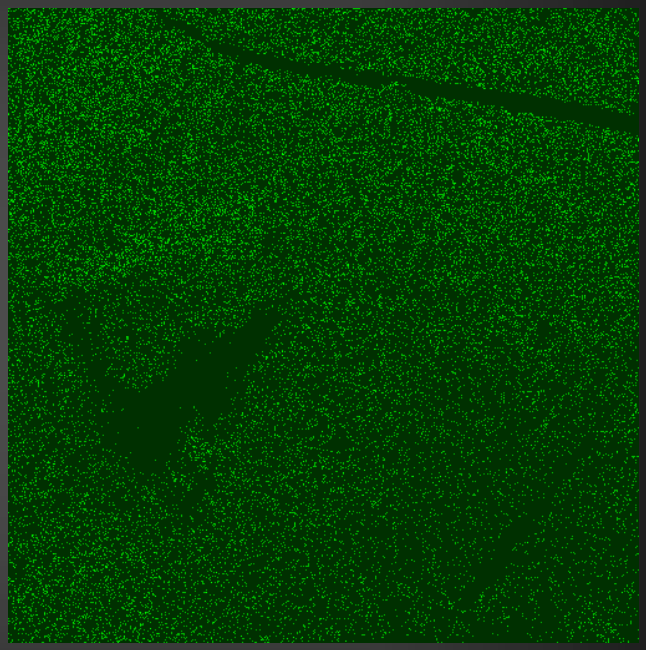
elemi részecske (R) = 3.0 nm
= 1.8 nm

korrelációs hossz (ξ) = 29 nm módosított formula
= 20 nm eredeti formula

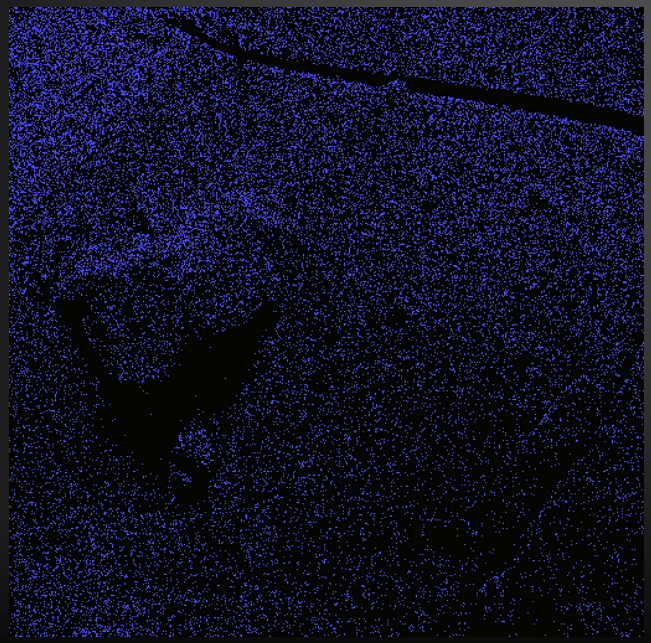
Mapping SEM



MAPPING Photo
AlOOH (×300)



AlOOH
(Al)



AlOOH (O)

Köszönöm a figyelmet!