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Mesostructured silica studied by silicon filtered reverse cross-polarization solid state NMR

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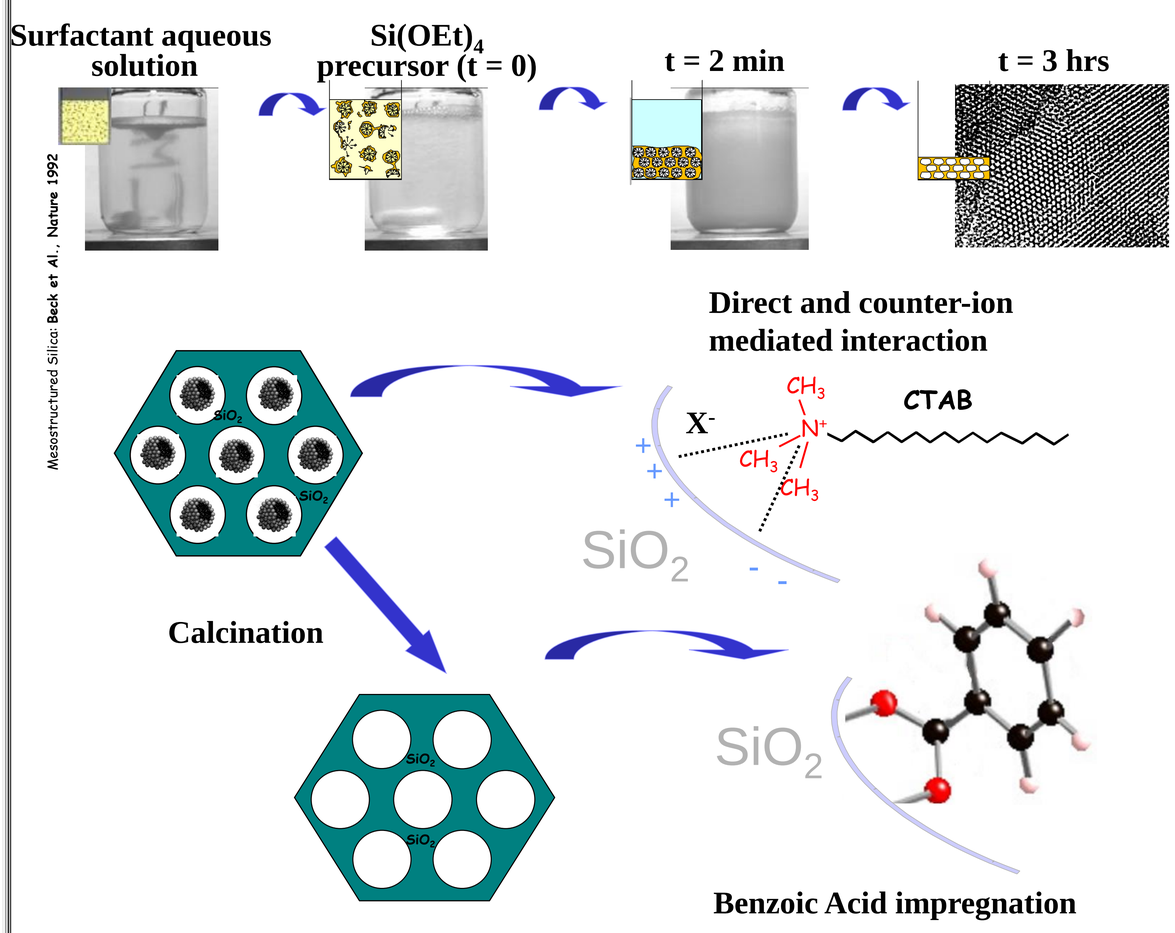
Nano-organised silica powders, prepared from self-assembly of surfactants and siliceous species, have done their breakthrough in the early '90s and, ever since, the interest of the material's community is growing continuously because of the extreme versatility of the process and the important number of possible applications achievable. Despite numerous contributions to the subject, some basic information concerning interactions at organic/inorganic interface has not been clarified yet. This poster will try to show how some advanced solid state NMR experiences can contribute to give more insights to some structural problems at the silica/surfactant interface and host-guest interactions between silica and embedded molecules.

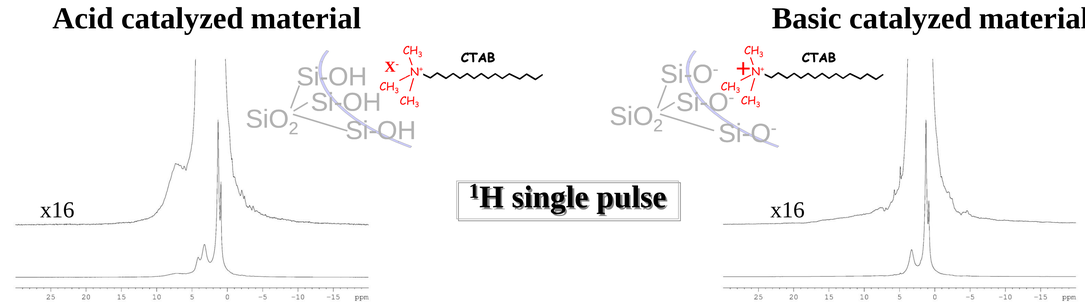
Introduction



$CTA^+ – SiO_2$ interactions

B_0= 7.04 T; MAS= 14 kHz

Acid catalyzed material | **Basic catalyzed material**

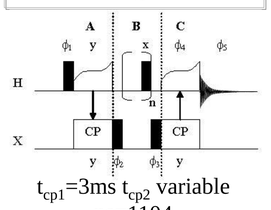


^{1}H single pulse

- CTAB signal stronger than silanol

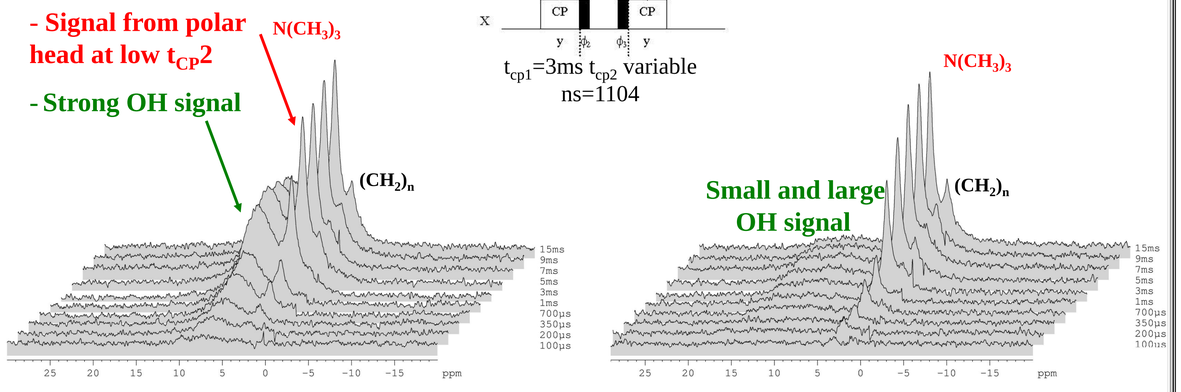
-No silanol signal

Reverse CP-MAS ^{1}H-^{29}Si-^{1}H



t_{cp1}=3ms t_{cp2} variable ns=1104

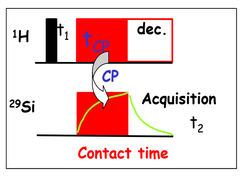
- Signal from polar head at low $t_{CP}2$
- Strong OH signal



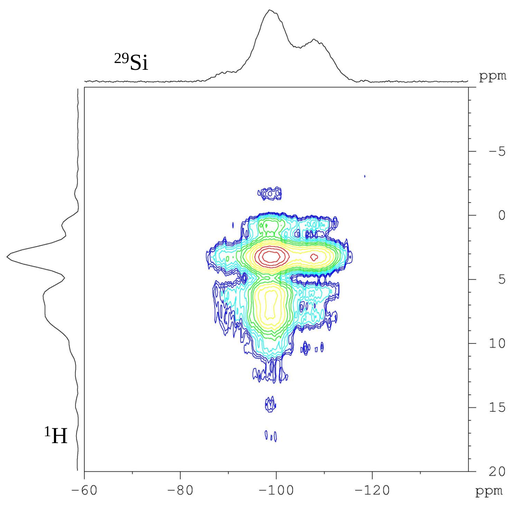
CTA^+ is further away from surface in acid-based materials

2D heteronuclear correlation

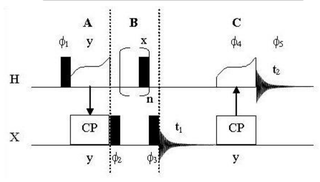
Normal hetcor ^{1}H-^{29}Si



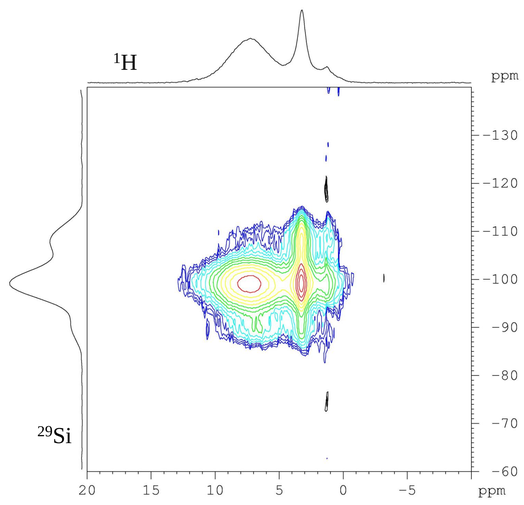
ns=800, td_1=64, si_1=256, t_{cp}=3ms, B_0=9.4T

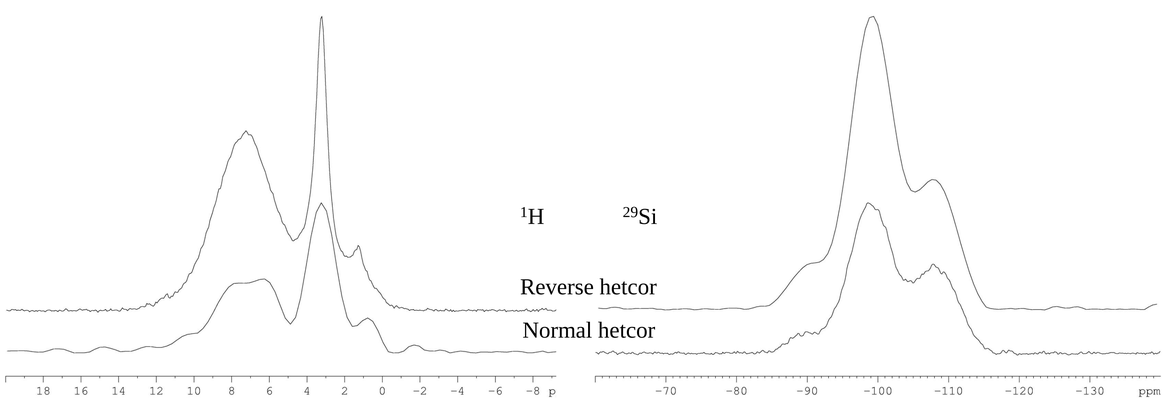


Reverse hetcor ^{1}H-^{29}Si-^{1}H



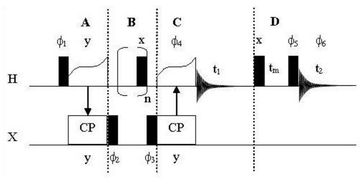
ns=800, td_1=59, si_1=256, t_{cp1}=t_{cp2}=3ms, B_0=9.4T



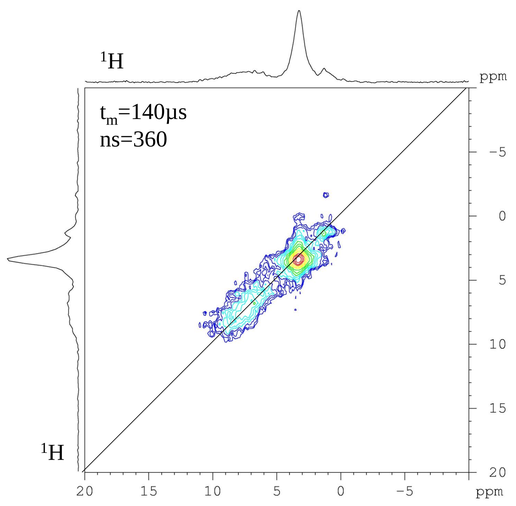


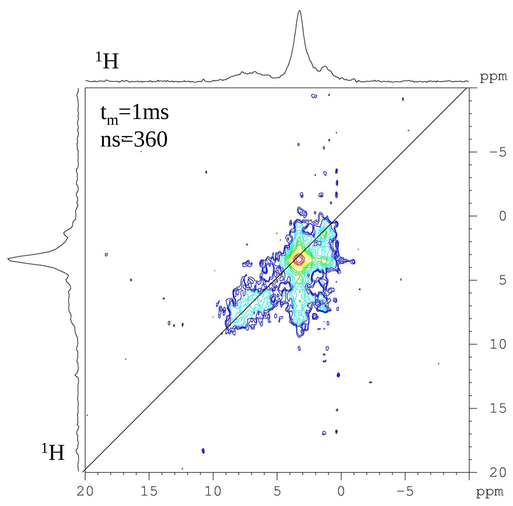
- **Signal enhancement**
- **Resolution enhancement**

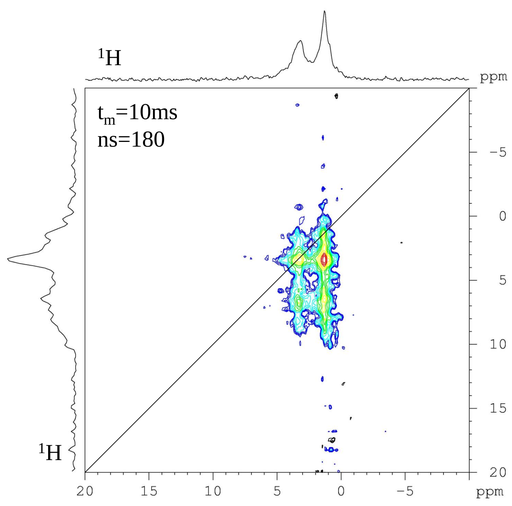
2D silicon filtered noesy



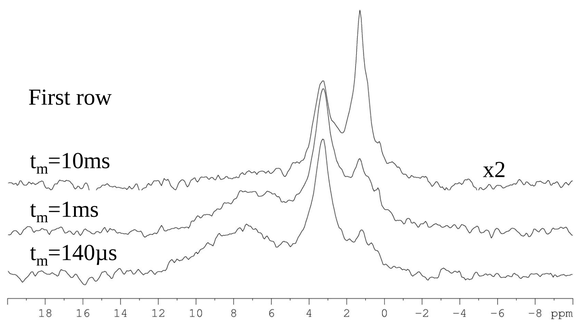
td_1=128, si_1=256, t_{cp1}=3ms, t_{cp2}=5ms, B_0=7.04T







- peaks on one side of the diagonal



- **Probing only ^{1}H close to silica**