

Efficient Hybrid Materials for Optical Power Limiting at Telecommunication Wavelengths

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Determination of sol condensation ratio by ^{29}Si NMR

^{29}Si NMR analysis was done on the sol as synthesized on a Bruker DRX400 (79 MHz for silicon NMR), using a capillary of acetone- d_6 as a reference. The calculations were done using MNova software.

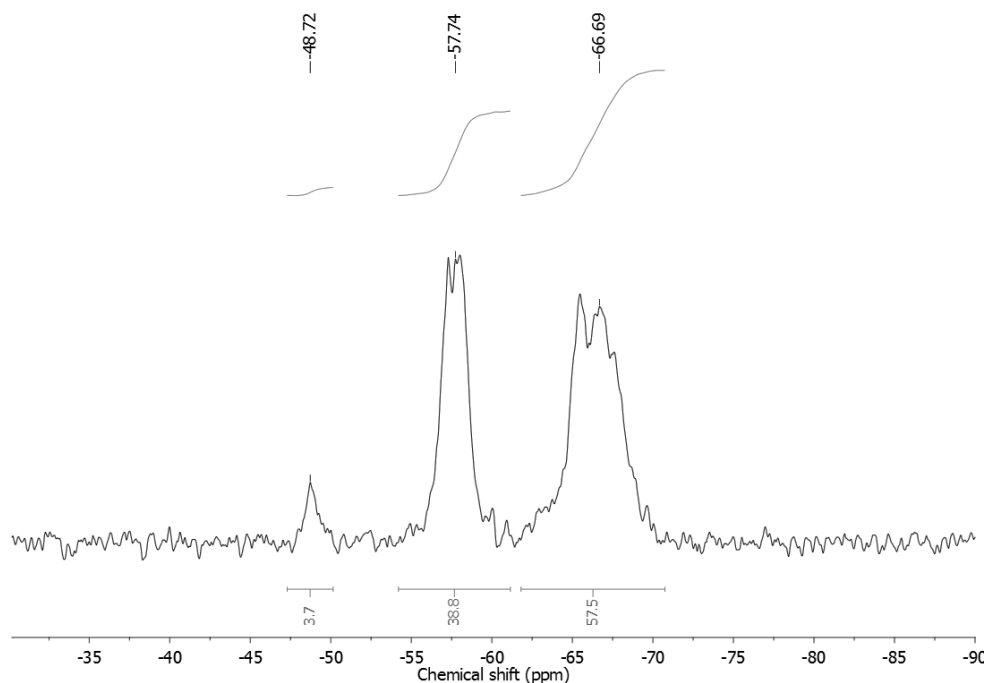


Figure S1. Example of ^{29}Si NMR for the MTEOS sol prepared using hydrochloric acid:

This sol of MTEOS is completely hydrolyzed and heavily condensed so only the T^1 , T^2 and T^3 populations (peaks at -49 ppm, -58 ppm and -67 ppm respectively in this case) are present.

The sum of all integrals was normalized to 100 to simplify calculations.

The condensation ratio (CR) can be defined by the ratio between the actual number of siloxanes bond in the sol and the maximum possible number of siloxanes bonds (3 possible

siloxanes bridges per silicon for MTEOS sols). It can be calculated using the relative area percentage of each population in ^{29}Si NMR:

Equation S2.
$$CR = \frac{1}{3} * \%T^1 + \frac{2}{3} * \%T^2 + \%T^3$$

With $\%T^1$, $\%T^2$ et $\%T^3$ the relative area percentage calculated from the NMR spectra.

In this case $\%T^1=3,7$ $\%T^2 =38,8\%$ et $\%T^3=57,5\%$ since no other peaks are present, so $CR=85\%$ for the hydrochloric acid catalyzed sol.

For the citric acid catalyzed sol, the condensation ratio was calculated the same way using the MTEOS and VTEOS peaks. No T^1 were present in this sol, overall $\%T^2= 30\%$ and overall $\%T^3=70\%$ so the CR was 90%.