New Expectation from DNP-Enhanced SS-NMR to figure out the Role of the Organic-Silica Interfaces: the Case of Diatom Frustules and Marine Siliceous Sponge Spicules

Sylvie Masse, Guillaume Laurent, Thibaud Coradin, Andrzej Pisera

To cite this version:
Sylvie Masse, Guillaume Laurent, Thibaud Coradin, Andrzej Pisera. New Expectation from DNP-Enhanced SS-NMR to figure out the Role of the Organic-Silica Interfaces: the Case of Diatom Frustules and Marine Siliceous Sponge Spicules. Magnetism and Magnetic Resonance: Magnetic Resonance, Understanding, Measurements and Modeling, Jun 2019, Strasbourg, France. hal-02156116

HAL Id: hal-02156116
https://hal.sorbonne-universite.fr/hal-02156116
Submitted on 18 Jun 2019

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Distributed under a Creative Commons Attribution - NonCommercial| 4.0 International License
New Expectation from DNP-Enhanced SS-NMR to figure out the Role of the Organic-Silica Interfaces: the Case of Diatom Frustules and Marine Siliceous Sponge Spicules

Sylvie MASSE 1, Guillaume LAURENT 1, Thibaud CORADIN 1 and Andrzej PISERA 2
1: Lab. Chimie de la Matière Condensée de Paris, Sorbonne Université, 4 place Jussieu, 75005 Paris, France
2: Institute of Paleobiology, Polish Academy of Sciences, ul. Twarda 51/55, 00-818 Warszawa, Poland

Microalgal culture at ISOMER: in collaboration with Dr Veronique Martin-Jezzil and Dr Benoit Tesson Faculté des Sciences et Techniques, Nantes, France

MNR Facility-Sorbonne Université Campus P. et M. Curie - T 32-33 SB

Living sponge collection: Pr A. PISERA on board of the IRD ship for an expedition in New Caledonia (Financially supported by National Science Centre, Grant No. 2016/21/B/ST10/02332)


References

While a lot of work is needed to figure out the organic-silica interfaces in natural materials such as diatom frustules or marine siliceous sponge spicules, Solid-State NMR appears to be a powerful toolbox with several nuclei and methods to carry out. Nevertheless, natural abundance in 2H as well a too poor C-content in the cleaned specimen do not allow nor 2D correlations neither well-resolved 1D spectra, that are necessary to go further on species proximity and connectivity assessment. Conjugating DNP to SS-NMR appears to be a promising solution to enhance the signal.

Conclusion

Variability in 14C CP-MASS-NMR response depending on species and history: nature of the taxon, aging, conservation, chemical treatment... (Ref.2).

Sensitivity limit

1H MAS and 29Si HPD-EDC-MAS NMR spectra of the Whole-cell, SDS-treated and H2O2-treated diatom frustules samples isotopically enriched in 2H, 15N, 29Si, and 13C, resp. (Ref.5). A signal broadening and a loss in intensity is observed after chemical treatment. While SDS/EDTA is used first to clean the frustules, further H2O2 treatment seems to be much more aggressive, probably leading to partial dissolution-recrystallization.

The two-valves silica cell wall (+ Frustule -) of diatoms: the silaffin-mediated formation of a silica shell embedded with organic matter.

The silica skeleton of sponges: the silicatën-mediated formation of a silica shell around an axial filiment, mainly composed of proteins.

Peak Signal-to-Noise Ratio (PSNR) and Root Mean Square (rms) for the 14C CP-MASS SS-NMR spectrum of the Thalassiothece diatom frustules and Laococetes Peronii (spoon spicules) samples. Signal and noise regions are highlighted with dotted vertical red and green lines, resp.; critical (Lc), detection (Ld) and quantitative (Lq) limits with dotted horizontal red, orange and green lines, resp. (according to ref. 3). In both cases, signal is detected but must be amplified.

Magnetic Resonance, Understanding, Measurements and Modeling – June 2-6, 2019, Strasbourg, France

3rd Thematic School: Magnetism and Magnetic Resonance