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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

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

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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/ST1/02332)

Conclusions

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 $^{13}$C as well as a too poor C-content in the clean specimen does 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.

References

1. Tessson B., Masse S., Laurent G., Maquet J., Livage J., Martin P., Thibaud CORADIN – 1H MAS and 29Si HPDEC-MAS NMR spectra of the Whole-cell, SDS-treated and H$_2$O$_2$-treated diatom frustule samples isopolitically enriched in $^{29}$Si, $^{13}$C, $^{31}$P and $^{28}$Si, resp. (Ref.1). A signal broadening and a loss in intensity are observed after chemical treatment. While SDS/EDTA is used first to clean the frustule, further H$_2$O$_2$ treatment seems to be much more aggressive, probably leading to partial dissolution-recrystallization.


Variability in $^{13}$C CP-MAS NMR response depending on species and history: nature of the taxon, aging, conservation, chemical treatment... (Ref.2).

Conclusion

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 $^{13}$C as well as a too poor C-content in the clean specimen does 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.

References


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