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Solid state NMR characterization of the waterlogged wooden part of Acqualadrone roman rostrum

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The roman rostrum found in the sea of Acqualadrone (ME) was characterized in our previous papers ^{1,2}. In the present work solid state NMR spectroscopy was applied on a wooden sample of the same artefact collected by coring and divided in four parts in order to correlate the conservation state to the depth. Results were compared with those obtained for a modern wood of the same species.

A structural study was performed by the acquisition of ¹³C Cross Polarization Magic Angle Spinning (¹³C CP MAS NMR) spectra. These spectra were acquired to assign the chemical shifts of the species that are present in the wooden matrix. In addition the spectra analysis allowed us to determine the cellulose crystallinity degree and the lignin condensation degree.

The holocellulose-lignin ratio and the cellulose-lignin residual interactions were evaluated through variable contact time (VCT) experiments and through the relaxation times determination respectively. Furthermore these latter experiments allowed us to evaluate the dynamic modifications occurring in the lignin and in the cellulose during the degradation process.

Solid state NMR results showed a decrease of carbohydrates with a complete cleavage of hemicelluloses. The crystallinity degree decreases in depth while the lignin condensation was not modified. Dynamic relaxation times studies demonstrated cellulose and lignin are still interacting to each other in the deepest samples of the wood.

[1] F. Caruso, M.L. Saladino, A. Spinella, C Di Stefano, P. Tisseyre, P., S Tusa, S. And E. Caponetti, *Archaeometry*, 2011, **53**, 547–562.

[2] P. Frank, F. Caruso, and E. Caponetti, E., *Analytical Chemistry*, 2012, **84**, 4419.