Comparison of loss-on-ignition and CHN-analyser methods for measuring sedimentary organic matter

Solinas G.¹, Braga F.¹, Sarretta¹, Matteucci G.², Molinaroli E.³, Botter M.¹, Guerzoni¹

(1)CNR - Istituto di Scienze Marine, Riva VII Martiri 1364/A, 30122 Venezia, Italy
(2)Gruppo CSA – Istituto di Ricerca – Via Al Torrente 22, 47900 Rimini, Italy
(3)Dip. Scienze Ambientali, Università Ca' Foscari Venezia, Dorsoduro 2137, 30123 Venezia, Italy

In order to detect the source of error in LOI determinations and to correct differences with CNH data, we analysed more than 70 sediment samples collected from the tidal flats of the Lagoon of Venice. Three different leaching procedures (LOI₃₅₀, at 350°C for 16 hours; LOI₄₅₀, at 450°C for 4 hours; and LOI₅₅₀, at 550°C for 3 hours) were analysed and compared with total organic carbon (TOC), total carbonates (TC) and grain-size composition.

Average values were 3% for LOI₃₅₀ (range: 0.5-13%), 5.5% for LOI₄₅₀ (range: 1.5-19%) and 12% for LOI₅₅₀ (range: 3-23%). TOC content measured by CHN analyser ranged from 0.2 to \sim 6%, and carbonates from 30 to 60%. Grain-size data for the sediment samples show a distribution ranging from slightly muddy sand to mud (with clay content varying from 3 to 46%).

The best fit between LOI and TOC values was found for LOI₃₅₀ (LOI₃₅₀=1.9*TOC + 0.78; r^2 =0.94), followed by LOI₄₅₀ (LOI₄₅₀=2.6 TOC + 2.5; r^2 =0.85), whilst a poorer correlation was found for LOI₅₅₀ (LOI₅₅₀=2.3*TOC + 9.1; r^2 =0.48).

Both the LOI_{450} and LOI_{550} values represent a significant overestimate, evident from the high values of the intercept when TOC is equal to zero. Following Frangipane et al $(2008)^1$, who found that loss of clay structural water at 350°C and 567°C corresponds approximately to 6-10 % of the clay fraction (<4 μ m), we applied different correction factors to compensate for this loss. We found that applying reductions of 5, 10 and 15% to the clay content improved the correlations for LOI_{350} , LOI_{450} and LOI_{550} respectively. As for the residual differences between the LOI_{450} and LOI_{550} values and those given by the CHN analyser, it was assumed that these may be accounted for by losses arising from the breakdown of carbonates. It was found that applying reductions to the TC content of 2-3 % in the case of LOI_{450} and 10-12 % in the case of LOI_{550} drastically increased the correlation between the LOI_{450} and LOI_{550} values and the TOC content as measured by CHN analyser.

-

¹ Gretel Frangipane, Mario Pistolato, Emanuela Molinaroli, Stefano Guerzoni, Davide Tagliapietra. Comparison of loss on ignition and thermal analysis stepwise. *Aquatic Conserv: Mar. Freshw. Ecosyst.* 19: 24–33 (2009)