Particulate Organic Matter (POM) in Suspended Sediments: Loss-on-Ignition Method Evaluated by Carbon Analysis on a Danube Standard

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Introduction: Suspended and fine grained sediment is one of the key water quality parameters in fresh and marine waters, affecting river-bed quality, transport of metals, nutrients and organic pollutants.
The flux of C occurs in both dissolved and particulate (particulate inorganic carbon, PIC; particulate organic carbon, POC) forms and the latter influences the sediment management.
The objective of this study is to examine the POM, determined by loss-on-ignition (LOI), and OC in suspended sediment "reference" material extracted from a specific recharge area and controlled by TGA and dry combustion analyser. The wide adoption of the LOI method reflects its ease of use, it is inexpensive, it is rapid, requires no specialized training.
Methods: From 168 L of Danube water 3.2 g of suspended sediment was obtained as "reference" material by settling, folded filter paper and drying at 105° C in Sept. 2015 (Fig. 1). Only 0.057 g (1.8%) were lost.

Fig. 1: Suspended sediment sampling from Danube water by a submersible pump.

The obtained "reference" material was analysed in duplicates for organic matter content by simultaneous thermogravimetry – differential scanning calorimetry and multi-phase carbon analyser, for specific weight (helium pycnometer) as well as mineralogy by x-ray diffraction.
This specific "reference" material was used to evaluate the procedure and the uncertainty of the determination of particulate organic matter (POM) by the loss-on-ignition method. The best results were obtained by the following procedure: On pre-heated (400°C) and pre-weighed glass fibre filter (*Whatman GE/F <0.7 µm*) a water sample with approximately 20 mg of suspended sediment is vacuum or pressure filtrated. After drying at 90°C (overnight) the filters with suspended sediment on top are reweighed carefully with a balance with an accuracy of ± 0.1 mg. After testing several temperatures and independent monitoring of the temperature (infrared thermometer) in the furnace the best heating temperature of 340±10° C is applied over 2 hours.
The sampling efficiency of glass fibre filters and cellulose membrane filters (MCE <0.45µm) was compared on Danube winter water in triplicates January 2017.
Results: The combination of thermogravimetry and carbon analyser result in a best estimate of particulate organic matter (POM)-content of 5.5 % (5.1-5.9%) in the "reference" suspended sediment. The specific weight of 2,43 g/cm^3 and the mineral content of quartz, calcite, dolomite, mica-illite as main components and chlorite, feldspar as minor components agrees with the analysed POM-content.
LOI-measurements of the "reference" suspended sediment deviate less than 0.5 wt.-% from the value obtained by thermogravimetry and carbon analyser. The CV of duplicate measurements are in the range of 6 – 9%.
The comparison of replicate samples of glass fibre filters and membrane filters (MCE <0.45µm) from the same sampling beaker in January 2017 resulted in an identical quantity of suspended sediment per litre (6.5 *vs.* 6.4 mg).
Discussion: Too high temperatures (>400° C) during the execution of the LOI-method caused in many studies the overestimation of POM. This may be due to the mineral structural water loss and destruction of carbonates at higher temperatures. A temperature around 340 °C was identified as optimum as it burnt most organic carbon, destroyed no inorganic carbon, caused less clay structural water loss [1][2]. Extracting a larger amount of suspended sediment in one catchment area as "reference" material allows to apply the LOI-method to suspended and fine grained sediments with smaller uncertainties.
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References: [1] Kralik (1999) *Applied Geochemistry*, **14**:807-816; [2] Salehi et al. (2011) *Pedosphere*, **21**:472-483.