



Assessment of methods for organic and inorganic carbon quantification in carbonate-containing Mediterranean soils

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Quantification of soil organic matter (SOM) stocks and fluxes continues to be an important endeavor in assessments of soil quality, and more broadly in assessments of ecosystem functioning. The quantification of SOM in alkaline, carbonate-containing soils, such as those found in Mediterranean areas, is complicated by the need to differentiate between organic carbon (OC) and inorganic carbon (IC), which continues to present methodological challenges. Acidification is frequently used to eliminate carbonates prior to soil OC quantification, but when performed in the liquid phase, can promote the dissolution and loss of a portion of the OC. Acid fumigation (AF) is increasingly preferred for carbonate removal, but its effectiveness is difficult to assess using conventional elemental and isotopic analyses. In addition, the potential effects of AF on SOM are not well characterized. The objective of the current study was to apply a multi-method approach to determine the efficacy of carbonate removal by AF and its effects on the residual SOM. We selected a set of 24 surface agricultural soils representing a large range of textures, SOM contents and presumed carbonate contents. For each soil, OC was determined using wet combustion (Walkley-Black) and IC was determined using the calcimeter method. Samples were then subjected to elemental (total C) and isotopic ($\delta^{13}\text{C}$) analyses by dry combustion using a Costech autoanalyzer coupled to a Thermo Finnigan Delta Plus isotope ratio mass spectrometer (IRMS) before and after AF. IC was equated to total C determined after fumigation, and OC was estimated as the different in total C before and after AF. Samples were also subjected to ramped oxidation using a Netzsch STA109 PC Luxx thermal analyzer coupled to a LICOR 820A infrared gas analyzer (IRGA). Quantification of OC was performed using evolved gas analysis of CO_2 (CO_2 -EGA) in the exothermic region 200-500°C associated with organic matter combustion. IC was quantified by CO_2 -EGA in the endothermic region 500-800°C associated with carbonate decomposition. Finally, changes in SOM composition or structure were assessed qualitatively through observed changes in the shapes of the exothermic regions of the CO_2 -EGA and DSC thermograms. Preliminary results of the thermal analysis indicate that acid fumigation is a highly effective method for soil carbonate removal, but also generates substantial changes in SOM composition/structure. Overall, our multi-method approach provided an excellent means of assessing methods for OC versus IC quantification in carbonate-containing Mediterranean soils.