Geophysical Research Abstracts
Vol. 17, EGU2015-13535-2, 2015
EGU General Assembly 2015

Highly resolved thermal analysis as a tool for soil organic carbon fractionation - methodological considerations

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Organic carbon (OC) in environmental samples consists of a continuum of molecules with different chemistry and turnover. Thermal methods provide a useful tool to differentiate OC fractions according to their activation energies. The higher the temperature needed for combustion, the higher the activation energy and the lower the energy-gain for microorganisms in the decomposition process. However, until now there is no method, which is able to quantify organic carbon fractions as well as total, organic and inorganic carbon in one analytical run. Here, we present methodological tests regarding effects of (1) ramp speed (12 vs. 35°C), (2) introduction of temperature plateaus (hold) for better peak separation and (3) sample amount, all of which potentially affecting results of thermal analysis. The used machine is a MCD RC-412 (Leco corporation) with highly resolved IR detection of CO_2 (3 times per second) during ramped combustion.
Regression analysis of the two ramp speeds showed, that the outcome of anlysis was not affected. The intercept was not significantly different from 0 (0.14 $\pm$3.15, p = 0.961) and the slope not significantly different from 1 (0.996 $\pm$0.0094, p = 0.969). A ramp speed of 35oC min-1 is preferred because of decreased analysis time. Performing analytical runs with and without holds showed again, that the intercept was not significantly different from 0 (-1.40 $\pm$ 1.14, p = 0.232) and the slope did not differ significantly from 1 (1.081 $\pm$ 0.042, p =0.067). Inclusion of a ramp increases confidence in results due to better peak separation. However, this was only tested for a range of different soils and care should be taken to transfer results to other environmental media and should be tested specifically for soil types not tested, yet. The amount of sample had some effect, especially when using more than 20 mg sample. Thus, sample amount shoulb be kept low, which calls for excellent homogenization of sample material. Overall, the MCD RC-412 with the tested setup is a suitable alternative for soil carbon analysis with a higher amount of information, as compared to bulk carbon measurements.