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Quantitative differentiation between coal, black carbon and soil organic matter in a minesoil matrix using thermal analysis techniques

Jaclyn Chan (1), Thomas Baumgartl (1), Peter Erskine (1), Clement Peltre (2), and Alain Plante (3)

(1) The University of Queensland – Sustainable Minerals Institute: Centre for Mined Land Rehabilitation, Brisbane, Australia, (2) University of Copenhagen, Department of Plant and Environmental Sciences, Copenhagan, Denmark, (3) University of Pennsylvania, Earth & Environmental Science, Philadelphia, United States

Minesoils are created during the rehabilitation of mined lands, typically covering newly constructed landforms. These soils are developmentally young, with little to no organic matter due to losses during stockpiling, disturbance and dilution from mixing with overburden material. Increasing soil organic matter (SOM) content in minesoils can improve soil quality for subsequent use, and thus SOM levels could potentially be used as an indicator of rehabilitation success. Assessment of SOM accumulation in minesoils from coal mines is problematic because of the varying amounts of coal dispersed in them. A further complication is the potential presence of a third carbon source, pyrogenic or black carbon (BC) derived from vegetation burning. Black carbon is nearly ubiquitous in Australian soils and especially in Vertisols, which are common in the Bowen Basin of Queensland, Australia, an active coal mining region. The quantification of SOM accumulation in coal minesoils is therefore complicated by the varying presence of both coal and BC. The objective of this study was to quantitatively differentiate between coal, BC and soil organic matter in a minesoil matrix as a means of assessing SOM accumulation during rehabilitation. Multivariate curve resolution – alternating least squares (MCR-ALS) is a chemometric deconvolution technique that makes it possible to decompose an unresolved mixture signal into several individual components and to estimate their concentration in the original mixture. We applied MCR-ALS to resolve curves generated during ramped combustion/oxidation. As a proof-of-principle, mixtures containing coal and/or BC (reference chestnut wood char) were created using three different matrices: sand, Vertisol and minesoil. Samples were analyzed using a Netzsch STA 409PC Luxx thermal analyser coupled to a LI-840 CO_2/H_2O infrared gas analyser (IRGA) for evolved gas analysis (EGA). MCR-ALS analyses were performed on area-normalized CO_2-EGA curves, using individual curves from BC, coal and soil as initial estimates for the pure components to be separated by the MCR-ALS.
Analyses of known mixtures yielded estimated proportions of coal with R2 = 0.87, BC with R2 = 0.76, and SOM with R2 = 0.99 confidence. The method was subsequently applied to the deconvolution of unknown samples in analyses of a set of rehabilitated minesoils. Results demonstrated high local heterogeneity of minesoils, where no consistent pattern in coal and BC contents were observed among different sites and soil depths. Overall, the results strongly support the proof-of-principle that thermal analysis coupled with MCR-ALS can quantitatively determine relative proportions of coal, BC and SOM in minesoils, and is a promising tool in assessment of minesoil rehabilitation.