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Chemical characterization of the black carbon reference materials – validating appropriateness based on elemental, spectral and isotopic analyses

K. Hammes (1), J.O. Skjemstad (2), R.J. Smernik (2) and M.W.I. Schmidt (1)

(1) Department of Geography, University of Zurich, Winterthurerstrasse 190, CH-8057 Zurich, Switzerland (2) CSIRO Land and Water, PMB 2, Glen Osmond, SA 5064, Australia (hammes@geo.unizh.ch / Fax: +41 1 635 6848 / Phone: +41 1 635 5183)

Black carbon (BC) is globally present as an inert residue resulting from vegetation and fossil fuel burning. BC has a diverse nature, and there are various methods with which to measure it (thermal, chemical, optical, indirect). An international ring trial was initiated to compare these different methods of analysis. For consistency, twelve reference materials were chosen as standard reference materials for BC measurement.

To validate if the reference materials were chosen appropriately to be representative of natural materials, we chemically characterised the twelve reference materials using elemental analysis, DRIFT, ^{13}C CP and BD NMR and δ^{13}C. The materials chosen for the ring trial can be split into two groups. The first group is expected to contain actual BC. It includes soot, charcoal (wood and rice), aerosol, two soils (chernozem and vertisol) and marine sediment. The second group (shale, melanoidin, natural organic matter from river water and two coals) was included to detect methodological artefacts. These materials are considered to contain little or no BC, but can possibly produce artefacts during application of some of the methods. Most of the reference materials were obtained through (standard material) suppliers, however, the wood char, rice char and melanoidin were produced by us. The wood char was produced by pyrolysis to simulate natural conditions in a burning log. No commercial standards for these materials are available. The starting materials for the wood and rice char were also chemically analysed.

The reference materials come from diverse environments, with large variations in C values, ranging from 2 % for the chernozem to 90 % for the soot. The clay percentage of the vertisol (37 %) is twice as high as the chernozem (19 %), which plays a significant role in the protection of organic matter and possible BC too. Soot had the lowest H/C and O/C ratios, indicating the presence of condensed ring structures. Both char materials had relatively low H/C ratios (thus hydrogen-poor) but enough oxygen functional groups to have a moderate O/C ratio. The uncharred wood and rice had high H/C and O/C ratios and plotted in the cellulose region as expected. The other standard materials had H/C and O/C ratios within the typical range for these materials given in literature. This elemental data is supported by the CP and BD NMR analysis done on the materials. Thermal treatment of the wood and rice material resulted in a loss of O-alkyl structures and an increase in aromatic carbon. The ^{13}C CP an BD MAS spectra for the vertisol and chernozem display two to three times more aryl C than any other structure, indicating a large presence of aromatic carbon. The δ^{13}C values of the materials range from –29.4 per mille for the shale to –16.4 per mille for the melanoidin. The δ^{13}C values of the uncharred wood (-26.6 per mille and uncharred rice (-26.4 per mille decreased only slightly (-0.3 per mille and -0.1 per mille respectively) during charring and might not be significant. Summarising, from the data presented it can be concluded that the reference materials represent typical natural samples and underscore characterisation data from literature, rendering them appropriately chosen for the black carbon ring trial.