



Optimizing instrumental parameters for Pyrolysis-GC/MS of peat

Kristy Woodard (1), Miriam Gross-Schmölders (2), Christine Alewell (2), and Jens Leifeld (1)

(1) Agroscope, Climate and Agriculture, Reckenholzstrasse 191, CH-8046 Zürich (jennifer.woodard@agroscope.admin.ch),

(2) Environmental Geosciences, University of Basel, Bernoullistrasse 32, CH-4056 Basel

Peatlands are the most widespread wetland type in the world, and have global importance for biodiversity, paleoecology, hydrological function, and biogeochemical cycling. Impacts of climate change as well as drainage for agriculture and forestry have resulted in widespread degradation in these sensitive ecosystems. Aerobic decomposition increases in degraded peatlands, shifting the carbon storage function from sink to source. Restoration efforts may not be considered effective unless overall net primary productivity exceeds decomposition, resulting in accumulation of peat and carbon stores.

Characterizing the molecular components within the peatland profile can delineate periods of degradation (decomposition) and restoration (accumulation), based on ratios of microbial matter to undecomposed plant material. Pyrolysis gas chromatography mass spectrometry (Py-GC/MS) has been suggested as an effective method for characterization of organic matter (OM) in peat without the need for time-consuming wet chemistry pretreatment techniques. While Py-GC/MS is a useful tool for molecular fingerprinting, challenges exist with this method of analysis. Secondary reactions may occur from cleavage of larger macromolecules, resulting in the formation of novel compounds and inhibiting fragmentation of others. Adjustments in temperature or heating rates may be needed for compounds of varying molecular weight and polarity, or to release OM bound to a mineral matrix.

Carbonaceous char left behind after pyrolysis typically retains inorganic components from the original sample, as well as highly recalcitrant organic compounds. The amount of OM in pyrolysis char is not necessarily proportionate to the amount of material volatilized, and can be impacted by instrument parameters, sample size, or the presence of minerals or metal cations in the original sample. Methods to improve transfer of pyrolysis products from the instrument to the GC column vary across the literature, and large variations in Py-GC/MS total ion intensities have been observed across different methods. A standard method of calibration to relate data from varied instrumental parameters is not yet available.

Chromatographic peak areas have been related to mass of the original sample to compare the effects of temperature and time on pyrolysis product formation, and some previous studies normalized pyrogram peaks by measuring sample weight before and after pyrolysis as a part of their methodology. However, while “yield” data for pyrolysis of biomass for production of biochar and bio-oil are found in the literature, changes in pyrolyzed sample mass or percentage of volatilized matter are infrequently reported in analytical studies. Fewer still are reported in studies investigating analytical pyrolysis of peat.

To better understand the capacity of Py-GC/MS analysis to characterize macromolecular components, the current study aims to determine the effect of instrumental parameters on remaining pyrolysis sample char. Samples are varied by mass, pyrolysis temperature, and particle heating rate. Peat samples with varied OM content have been selected to investigate the effects of mineral components on efficiency of peat pyrolysis. Elemental analysis is conducted on the char residue of pyrolysis samples to determine carbon: nitrogen ratios and method reproducibility.