Analysis of Nonvolatile Soil Lipids by Curie-Point Pyrolysis. Revisiting the Environmental Information Gained from Soil Biomarker Assemblages

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Soil lipids have been for a long time recognized as a first order source of environmental information. In fact, soil lipid molecular assemblages are highly responsive for recent changes in the structure of the trophic system and also represent a valuable agrochemical record of environmental changes in the past.

Conventional analysis of soil lipids can be easily performed by non-destructive methods such as gas chromatography-mass spectrometry (GC/MS) applied to soil extracts obtained with suitable organic solvents and derivatized to achieve maximum volatility. Nevertheless, several recent experiments have evidenced that the molecular information provided by the soil lipid fraction is far to be completely utilized. In fact, a series of quantitative studies on lipid extracts from forest and cultivated Mediterranean soils has shown that up to 50% of the total lipid remain "invisible" under "normal" chromatographic conditions. It is clear that most of this material is non-volatile or probably remain retained in the first few cm of the capillary column.

Some progress has been achieved in the last years concerning the above limitation. In particular, i) forced chromatographic conditions with ultra-high-temperature metallic columns have indicated that some of the non-detected material consists of long-chain alkyl compounds, ii) the alternative use of fast chromatography in very-short (<3 m) columns have evidenced the occurrence of additional soil lipid components, such as wax esters, polyditerpenes and esteryl esters and, iii) the use of high-resolution ^{13}C NMR on whole lipid samples showed complex aromatic/unsaturated signals including isoprenoid compounds such as polyprenols and probably complex cycloalkane structures.

The analysis of soil lipids by Curie-point analytical pyrolysis (Py-GC-MS) showed some common features with the results from direct injection indicating that rapid thermoevaporation of a large proportion of the lipid material occurs, but also an unexpectedly high amount of new structures not observed in the nonpyrolysed volatile fraction were observed. The additional pyrolytic molecular assemblages are interpreted as coming from fragmentation of a macromolecular or oligomeric lipid moiety probably formed from resinification of lipid reactive compounds in addition to products derived from the high-range homologues of alkyl compounds.

These preliminary results, which we intend to validate in a collection of lipid samples from unaltered or disturbed soils from Central Spain, indicate that analytical pyrolysis may provide enhanced biogeochemical information. The results are especially promising in the case of those lipid samples yielding cyclic biomarker structures (i.e., presumably not artifacts) by Curie-point pyrolysis.