MONITORING BY SOLID PHASE MICROEXTRACTION (SPME) THE DYNAMICS OF VOLATILE COMPOUNDS RELEASED FROM SOILS AMENDED WITH FOREST AND AGRICULTURAL WASTES

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Solid Phase Microextraction (SPME) combined with GC-MS is a recent and promising tool for the rapid analysis of high—and medium—volatile compounds occurring in gas, liquid and solid samples (Arthur and Pawliszyn, 1990), which is widely used in monitoring environmental pollution, in the analysis of biological and food samples (Pawliszyn, 1997, 1999) and in other areas such as agricultural research. Recent attention has been paid to the effect that volatile and non-volatile compounds present in—or released during—the decomposition of organic material could have on soil biota dynamics. In this work, SPME followed by GC-MS has been assessed for routine analysis of molecular assemblages' dynamics in the soil gas phase at different stages after soil treatment with agricultural and forest wastes used to improve their physico-chemical characteristics and for the control of crop pests and pathogens in agricultural soils.

Material and methods

Two experiments were carried out applying different organic amendments to soil moistened at field capacity and covered with a polyethylene sheet to retain the gases for 28–35 days. The experimental design included: 1) pine (*Pinus halepensis* Mills.) stems and leaves on Calcic Entisol (Játiva, Alicante, Eastern Spain), and 2) liquid sugar beet vinasses on Haplic Arenosol (Jumilla, Murcia, SE Spain). At different times the moist soil samples were collected and stored in closed flasks, and after equilibrium the flask headspace was analyzed by SPME followed by GC-MS.

Results and discussion

The results from the experiment with pine stems and leaves showed major peaks of mono- and sesquiterpene hydrocarbons such as caryophyllene, β-myrcene, *p*-cymene, selinene, β-terpinene, etc. In the case of untreated soil in experiments with sugar beet by-products up to six major compounds were found, i.e., cyclohexanone, limonene, butanone, acetic acid, camphor and benzaldehyde, whereas after inputs from the sugar beet there was a large concentration of short-chain alkyl compounds (mainly acids and alcohols) such as methylbutanal, acetic acid, butanediol and butanoic acid.

Our results showed that the gas phase composition of soil changed significantly with regard to the depth, the major compounds being xylenes and cyclohexanol at 20–30 cm level (Fig. 1A), nitrobutane and benzaldehyde at a depth of 40 cm, sulphur compounds such as methyl-, dimethyl- and trimethyldisulphide at a depth of 65 cm (Fig. 1B), and acetic acid and benzaldehyde at 85 cm depth. This suggested a large influence of water saturated zones of the soil in the composition of their atmosphere.

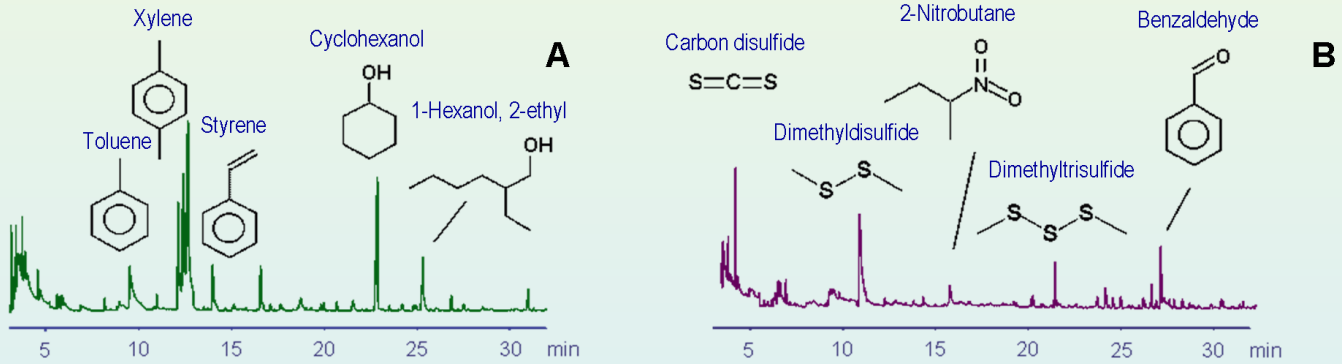

Figure 1. Solid Phase Microextraction GC-MS, total ion current profiles from the gas phase of soils treated with sugar beet industrial residues under polyethylene sheet. A: a soil depth of 20–30 cm, alkylbenzenes and aliphatic alcohols. B: underlying horizon (65 cm soil depth), sulphur and nitrogen compounds, aromatic aldehydes.

Conclusions

Solid Phase Microextraction represents an interesting tool that allows the fractionation of volatile compounds released by organic amendments applied to soil for their further analysis by GC-MS. The SPME GC-MS results showed large differences in the composition of the soil gas phase in accordance with the type of organic wastes and in terms of soil depth. Furthermore, SPME followed by GC-MS provides useful information to be employed in studies on the chemical factors controlling the soil biota dynamics in agro-ecosystems. Further research is required to validate the quantitative aspects of the method and to establish causal relationships between the molecular structure of the compounds identified by SPME GC-MS, as well as their interest in control of pathogenic organisms in agricultural soils.

References

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