Research Articles

Phytovolatilisation of Organic Chemicals

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Abstract

Background, Goal and Scope. When contaminated soil and water are remediated by using plants, a multitude of pathways, partly not yet quantified, are activated. 'Phytovolatilisation' denotes the process in which usually organic compounds are emitted from the aboveground sections of the plant into the atmosphere. This includes all the compounds which are taken up by the roots, partly transformed within the roots, and transported into the shoot.

Even though the elimination of potential environmental pollutants by plant uptake was estimated to be much lower than microbial degradation in the soil back in the 1970s, in the 1990s the phytovolatilisation of volatile pollutants became a topic of economic interest since it held out the promise of accelerating the elimination of pollutants during the remediation of contaminated sites. In the past, investigating phytovolatilisation often failed because of the analytical methods' high detection limits. More sensitive methods such as radioactive labelling usually only detected the cumulative elimination of pollutants by evaporation and provided little information on the mechanism of these emissions. Therefore, the experimental goal of this work was to determine the dynamics of specific emission rates.

Features. Independently of the volatilisation of the contaminants from the nutrient solution, the emission rates were determinated under defined, approximated natural conditions by using a dynamic gas exchange chamber. To detect traces of contaminants in air samples with high relative humidity, a purge & trap technology was used.

An estimation of the specific emission rates of different materials was made with a transportation model. The model was used to ascertain what compounds are most likely to be emitted by plants due to their physical characteristics.

Results and Conclusions. In experiments with 2,6-Dimethylphenol and Trichloroethylene, a clear link was observed between contaminant emission and the lighting intensity likely to be due to the stomata aperture. The absolute values of the emission rates were very low and in the range of nmol/h m² foliar surface. The calculation of the emission rates in different scenarios shows lowly higher emission rates for materials with lower n-octanol-water partition coefficients

Recommendations and Outlook. Phytovolatilisation is particularly suitable for eliminating volatile compounds in shallow groundwater contaminations, but the experimental proof of an increased net emission of planted areas with volatile soil contaminants near the surface compared with unplanted areas is still pending. On the basis of the model computations it is rather to be expected that the net emission of volatile lipophilic materials should be reduced into the atmosphere by using plants.

Keywords: Emission of VOCs; gas-exchange experiments; organic compounds; phytoremediation; phytovolatilisation; volatile organic compounds (VOC), Trichloroethylen, 2,6-Dimethylphenol

Abbreviations and variables: *TSCF*: Transpiration stream concentration factor; K_{ow} : n-Octanol/water partition coefficient; K_{AW} : Air/water partition coefficient

Introduction

Even though the elimination of potential environmental pollutants by plant uptake was estimated to be much lower than microbial degradation in the soil back in the 1970s (Felgner et al. 1967, Meissner and Friedmann 1968), the phytovolatilisation of volatile pollutants in the 1990s became a topic of economic interest since it held out the promise of accelerating the elimination of pollutants during the remediation of contaminated sites. In the past, investigating phytovolatilisation often failed because of the analytical methods' high detection limits. More sensitive methods such as radioactive labelling usually only detected the cumulative elimination of pollutants by evaporation and provided little information on the mechanism of these emissions.

Therefore, the experimental goal of this work was to determine the dynamics of specific emission rates. If the atmosphere is regarded as an additional compartment in the plantsoil system, the following distribution and transportation processes need to be considered (Fig. 1):

- A) The distribution of chemicals between soil solution and root tissue
- B) The transport of solutes from the root into the stem and leaves by the transpiration flow
- C) The decline in concentration of the chemicals taken up in the plant tissue by growth, transformation, immobilization and degradation
- D) The emission of volatile chemicals from the leaf surface (phytovolatilisation)
- E) The distribution of chemicals between leaf and atmosphere, especially the uptake of soil-borne chemicals by leaves



Fig. 1: Subprocesses of the transfer of pollutants from contaminated planted soil into the atmosphere

1 Material and Methods

1.1 Gas-exchange chamber

Due to the low emission rates and the disruptive influences of other emission sources, phytovolatilisation can only be measured quantitatively in prepared chambers. The experimental difficulties resulting from keeping living plants in closed chambers are known (Kreeb 1990). Studying trace emissions makes the experiments even more time-consuming.

The gas exchange chamber designed for these investigations consists of a split glass container (height 1.20 m, diameter 20 cm) in which the plant is separated from ambient air and cultivated as a hydroponic culture in a nutrient solution (Fig. 2).



Fig. 2: Dynamic gas exchange chamber with continuous dosage of contaminated nutrient solution

To distinguish between emissions from leaves and contaminated soil, the phytosphere and the root within the contaminated nutrient solution have to be separated. In the case of non-woody plants this airtight seal cannot be achieved by mechanical pressure. Therefore, an attempt was made to separate leaf and root by using a modified gas flow. The container for the roots was filled up with glass beads. The plant was covered between stem and root by a paraffin layer, which is borne on the glass beads and does not come into direct contact with the contaminated nutrient solution. The addition of liquid paraffin lowers the melting temperature of the paraffin wax mixture, resulting in a highly liquid melt at 40°C. The contraction of the cooling melt results in narrow columns remaining in the paraffin layer. A small stream of air can flow through these columns and thus thwart any convective flow of contaminants from the rhizosphere to the phyllosphere. The nutrient solution in the root container was constantly recirculated in order to mix it with fresh contaminated solution.

Purified air was passed through the upper section of the exchange chamber containing the leaves (Fig. 3). This stream of air is purified by catalytic oxidation and dried by passing it through a zeolite bed. The glass surfaces of the upper section were silylated to reduce adsorption effects. Our own work and that by Orchard (Orchard et al. 2000), for example, indicates that measurement artefacts can occur if low wind velocities are used in the exchange chamber because of the influences of the boundary layer and the contaminant



Fig. 3: Influence of wind velocity on total conductance for the water vapour of a leaf with constant conductance of the stomata and cuticle. a) 100% corresponds to total conductance without boundary layer resistance

concentration in the air. If the main wind direction is along the leaf longitudinal axis, the boundary layer conductance can be estimated (Gates 1980, p. 335).

To minimize the danger of artefacts measuring the rate of phytovolatilisation, the air was recirculated in the upper section using an external diaphragm compressor (KNF Neuberger, Germany, 100 L/min, 0.5 kW). A linear wind velocity of 0.06 m/s was reached – about 20% of the wind velocity measured in a plant stock at a reference wind velocity of 1.05 m/s over the plants (Ophoff 1998).

1.2 Chemical analysis

Because of the dilution of the phyllospheric air with purified air, the concentration of the contaminant is expected to be extremely low, necessitating an enrichment step for further analysis. Enriching the contaminants is hampered by the high relative humidity of the chamber air of 40-80%. Traces of organic contaminants must be separated from the main components in a number of steps. Since the herbal tissues are very reactive, metabolites may also be emitted. In order to be able to detect as broad a spectrum of compounds and concentrations as possible, enrichment with non-specific, solid adsorbents is the method of choice. Using a thermal desorption procedure enables automated analysis in order to observe the day/night dynamics of the emission rate. The Preconcentrator 7000 analyser (Entech Instrument Inc., California, USA) was used for automated sampling and enrichment. Since desorption heating was limited to 250°C, only Tenax[®], a porous polymer based on 2,6-diphenylphenol, could be used as adsorbent. During the selective multistage enrichment of the trace components, the adsorption conditions can be arranged largely independently of the requirements of the accumulative sampling procedure.

The flow chart is shown in **Fig. 4**. The 'modules' refer to chambers containing traps which are cooled with liquid nitrogen or heated by electrical heating or heated air.



Fig. 4: Diagram of the Preconcentrator connected to a gas chromatograph

A sample of chamber air is sucked in through trap 1 filled with glass beads by a vacuum pump. The volume of sampled air is controlled by a mass flowmeter. The module is cooled with liquid nitrogen to -150°C, so that all the components are frozen out with the exception of nitrogen, oxygen and the noble gases. After this step, trap 1 is flushed with cooled helium (-150°C) in order, above all, to remove oxygen. After closing trap 1 with an 8-way valve, the temperature in module 1 is raised to above 0°C (e.g. 20°C) so that the main component water is in a liquid state. The components with a higher dew point are volatile; after changing the valve position they are stripped with a defined helium stream and transferred into the next trap filled with adsorbent. After adsorption in trap 2, the components are desorbed at a higher temperature (e.g. 200°C) and again focused in the cooled trap 3 (e.g. -180°C). To inject the sample into the capillary column, trap 3 is heated within a few seconds to approximately 80-120°C with hot air. Multistage enrichment has the following advantages:

Table 1: Test condition

- Water (the main component) is drastically reduced.
- The adsorbent never comes into contact with the reactive gases oxygen, ozone or nitrous gases.
- Irrespective of the humidity of the original air sample, the adsorbent is loaded with the same quantity of water. The displacement of trace components by the water matrix ought to be reproducible.

The measurements and also rough calculations showed that if an optimised enrichment method is used, polar organic materials such as 2,6-dimethylphenol are also recovered to 95%, while the amount of water is diminished to 2%. The sample was analysed in an HP 6890 gas chromatograph (Hewlett Packard) connected to a quadrupole mass spectrometer (GC/MS system). The measuring instrument was calibrated with a permeation system.

1.3 Experimental programme

The emissions of exposed plants were examined in two test series. Marsh plants (helophytes) were selected due to their ability to survive in dammed anaerobic soils and their specifically high transpiration rates. The plants were exposed by way of example to 2,6-dimethylphenol (polar, low-volatile) and the chlorinated hydrocarbons trichloroethene, chlorobenzene and 1,2-dichlorobenzene (less polar, but highly volatile; **Table 1**).

2 Results

2.1 Exposure of Mentha aquatica to 2,6-dimethylphenol

At the beginning of the experiment, 317 mg 2,6-dimethylphenol (26DMPh) was dissolved in 30.8 L of a nutrient solution. This contaminated nutrient solution was pumped through the root vessel at a rate of 200 mL/min to avoid gradient of the 26DMPh concentration. After contamination, the plant was illuminated for 1 h at approx. 90 µmol/s m²; this was followed by the ninehour night phase. The emission rate in the dark phase after the beginning of exposure averaged 0–3 nmol/h m². An abrupt rise in the 26DMPh concentration in the phyllospheric air to about 60–80 nmol/h m² was observed the next day after just 6 hours

Plant	
<i>Mentha aquatica</i> Water mint	<i>Iris pseudacorus</i> Yellow iris
2030	4292
26DMPh	TCE, CB, 12DCB
10.3 mg/L at the beginning	200–500 μg/L
One-off (batch experiment)	Continuous
7.5	14.0 ^a / 4.5 ^b
9.6	100
7:00–22:00	7:00–22:00
9:00-20:00	9:00–20:00
12	20
	Mentha aquatica Water mint203026DMPh10.3 mg/L at the beginningOne-off (batch experiment)7.59.67:00-22:009:00-20:0012

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Fig. 5: Emission of 26DMPh after contamination of the rhizosphere



Fig. 6: Phytovolatilisation of 26DMPh of an exposed *Mentha aquatica* as a function of the 26DMPh concentration in the rhizosphere

of lighting (Fig. 5). In contrast, the emission of water (transpiration) rose directly when the artificial lightening was switched on. This effect may be due to the faster uptake of water by the plant in comparison to 26DMPh. The slow rise in the 26DMPh concentration in the contaminated nutrient solution at the beginning of the experiment may have the same cause. The average emission rates changed with the 26DMPh concentration in the nutrient solution (Fig. 6).

2.2 Exposure of Iris pseudacorus to chlorinated hydrocarbons (CHC)

In connection with the residual CHC pollution of a disused chemical industry site, measurements with the pollutants trichloroethene (TCE), chlorobenzene (CB) and 1,2-dichlorobenzene (12DCB) were performed. Two litres of a fresh nutrient solution were spiked with 10 μ L of TCE, CB and 12DCB each and treated in an ultrasonic bath for 1 h. This contaminated nutrient solution was injected continuously into the circulating nutrient solution so that, in the event of excess dosage, the CHC concentration ought to remain constant. The CHC concentrations in the rhizosphere, determined by means of SPME¹, were below the initial concentrations because of gaseous discharge with the air from the root container. Due to an undesigned contamination of the



Fig. 7: TCE emission from Iris pseudacorus (1st experimental phase)

fresh air with about 1–3 pmol/L CB and 12DCB, only the emission of TCE could be reliably observed. The emission maxima of TCE exhibited a periodicity of 24 h synchronous with the beginning of the light phase (**Fig.** 7).

The second experimental phase was designed to monitor the kinetics of the emission rate while interrupting the continuous contamination of the nutrient solution.

Due to the failure of the turbo-molecular pump of the mass spectrometer, the measurements had to be continued with a less sensitive heat conductivity detector under changed flow conditions in the gas exchange chamber. Thus, the absolute emission rates of the first and second experimental phases are not comparable (cf. Fig. 3).

Surprisingly, the CHCs were still emitted after the cessation of contamination. TCE in the nutrient solution was no longer detectable after 30 h (corresponding to exposure of 300 h), the remaining CHCs after 100 h (corresponding to exposure of 370 h). As the TCE emission rate fell after the end of contamination, the emission rates of CB and 12DCB constantly rose (Fig. 8). In comparison to the emission of 26DMPh, another source of contamination must be responsible for this continued emission of CHC. In order to rule out other possible emission sources, especially the paraffin and the sand layer, the above-ground plant parts were removed.



Fig. 8: Emissions after the cessation of the contamination of the rhizosphere (2nd experimental phase)

¹ Solid Phase Micro Extraction (Chai et al. 1993)



Fig. 9: TCE emission rate with intact plant and after cutting the leaves

About 24 hours after cutting, the emission rates were measured again. The values were based on the original leaf surface. Immediately after cutting the leaves, an abrupt rise in the CHC emission rate over the maximum daily values of the plantoccupied chamber was registered. However, within 17 h the emission rate dropped below the specific emission rate during the dark phase (**Fig. 9**). As the light phase began, no rise in the CHC emission rate was observed in the vacant chamber. The TCE emission reached a plateau at approx. 2 nmol/h m² despite the temperature rise in the chamber. The periodic change in the emission rate is therefore not due to the temperature-influenced desorption of the CHCs from a possible contaminated paraffin layer.

Instead, the initial rise in the TCE emission rate indicates that removing the above-ground plant parts opened up a source of contamination in the remaining parts. Given the extremely low CHC concentration in the nutrient solution, the sap in the conductive tissue leaking from the leaf stumps ought only to account for a tiny fraction of the CHC emission observed. Since the CHC emission subsided relatively quickly, a limited CHC pool might be the reason for this emission pattern. It is possible that the lipophilic, volatile CHC in the root was adsorbed and, in a desorption step, entered the air-filled aerenchymatous tissue.

3 Estimation of the Phytovolatilisation Rate

Model calculations were performed to identify materials with which particularly high phytovolatilisation rates should be expected. A clear model based on the material's physicochemical material parameters as well as the physiological parameters of the plant was developed by Trapp and Matthies (1995). In this model, the above-ground plant parts are simplistically assumed to be a single compartment, so that an analytical solution exists for the mass balance equation. This one-compartment model is a simplification of an existing four-compartment model (Trapp et al. 1994). In the model, the temporal change in the pollutant quantity in the above-ground parts of the plant is calculated using a differential equation. On the basis of this one-compartment model by Trapp and Matthies, the emission rates for selected materials were estimated.

The emission rate can be calculated from the equilibrium concentration of the contaminants in the leaf (Baeder-Bederski 2001). To elucidate the influence of the physico-chemical characteristics of the selected materials on the emission rate, the calculation was first performed for a constant concentration in the nutrient solution of 8.0 E-5 mol/L and a specific transpiration rate of 1.8 mol/h m² for an uncontaminated atmosphere neglecting metabolisation and growth. Under these conditions the emission rate in the model is affected only by the uptake rate of the chemical by the plant (scenario 1, cf. Fig. 10).

Sorption on the organic soil matrix reduces the availability of the materials for the plant. Whereas for the preceding scenario a carbon content in the soil of 0 Ma% was assumed, a carbon content of 2 Ma% is assumed in the next scenario (scenario 2, cf. Fig. 10).

In the soil-plant system, organic materials can be metabolised. This important step cannot be fully taken into account since it cannot be exclusively described by material parameters. Concerning phytovolatilisation, a decrease in the emission rates will have to be expected for the most part since xenobiotic compounds are immobilized into the vacuoles. However, this metabolisation enhances phytovolatilisation in some cases. For example, volatile dimethylselenide and dimethyldiselenide predominantly formed microbially from selenites and selenates can be emitted from the leaf surface (Beath et al. 1935, Lewis et al. 1966, Terry et al. 1992, Terry and Zayed 1994, Bañuelos et al. 1997, Hansen et al. 1998).

For this calculation, an experimentally determined metabolisation rate for 26DMPh of 0.08 h^{-1} is assumed for all materials (Baeder-Bederski 2001). The simultaneous metabolisation of these materials in the soil remains neglected (scenario 3, cf. Fig. 10).

The phytovolatilisation rate is also affected by the thermodynamic equilibrium between the leaf compartment and the atmosphere. In the next scenario, the contamination of the atmosphere (phyllosphere) by the soil-borne emission is therefore considered. This emission depends on specific characteristics of the materials as well as the soil structure and the boundary layer resistance above the soil (Jury et al. 1990). Jury and coworkers calculated for example a maximum benzene emission² of approx. 5,300 nmol/h m² from sandy soil and 5 nmol/h m² from clayey soil. Thus, the fraction of the soilborne emission of evaporation is not negligible. The emission rates of phytovolatilisation are reduced by soil-borne emission due to the changed position of equilibrium. Greatly simplifying this process, the concentration of contaminants in the air was calculated according to the relative evaporability of the materials from aqueous solution on the basis

² Related to the soil surface from a depth of 1 m with a benzene concentration of 3.2 E-4 mol/L

Scenario 1	Scenario 2
'Uninhibited emission'	'Sorption on the soil matrix'
The materials are only taken up as a function of the transpiration stream coefficient and emitted unmodified by the plant	Before uptake by the plant the materials are adsorbed on organic soil components
Scenario 3	Scenario 4
'Metabolisation and growth'	'Contaminated atmosphere'
The materials are additionally metabolised in the plant as non-volatile materials	Other emission sources in the atmosphere reduce emission by phyto-volatilisation

Table 2: Overview of the scenarios applied



Fig. 10: Phytovolatilisation rates in different scenarios (cf. Table 2)

of a typical benzene concentration³ of $10 \mu g/m^3$ (scenario 4, cf. Fig. 10). The results of the simulation are shown in Fig. 10.

The 'uninhibited emission' scenario features the most favourable - albeit unrealistic - conditions concerning phytovolatilisation. If the sorption of the materials on the organic soil matrix is considered (scenario 2), the phytovolatilisation rate decreases as in the case of 246TCPh from approx. 1,000 nmol/h m² to 10 nmol/h m². Irrespective of their evaporability and other transportation characteristics (in particular the TSCF) of the materials, the emission rate is reduced by sorption processes in the soil if the n-octanol-water partition coefficient of the material rises. The other scenarios influence the computed emission rate only in the case of less volatile materials ($\lg K_{AW} < -3$). No phytovolatilisation ought to be detected if the free concentration of the contaminant in the leaf decreases by metabolisation (scenario 3) and the leaf-air partition coefficient of the material is high (e.g. 246TCPh, cf. Fig. 10).

4 Conclusions

The phytovolatilisation rates were determined by measuring the current concentration of contaminants in the air of a gas exchange chamber so that the changes in the emission rates

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occurring with lighting intensity could be observed. To determine the organic trace components in the chamber air, two multistage procedures were applied based on selective enrichment on an adsorbent followed by thermal desorption. The daily changes in the emission rates can be attributed to the changes in light irradiation and the stomatal aperture. This was confirmed by emission measurements after a complete leaf cut.

Owing to the faster evaporation from the contaminated nutrient solution and the associated low contaminant concentration in the rhizosphere, the emission of trichloroethylene from an exposed *Iris pseudacorus* amounted to just a few nmol/h m². The phytovolatilisation of chlorobenzene and 1,2-dichlorobenzene was only qualitatively detected due to increased underground emission and the uncertain determination of the root zone concentration. In contrast to the also investigated 26DMPh emission of a water mint (*Mentha aquatica*), the emission of chlorinated hydrocarbons reacted only slowly to a change in the concentration of the soil solution. It can be assumed that these materials are adsorbed on the roots and also slowly emitted from intact plants through the aerenchymatous tissue.

In order to simulate the influence of the subprocesses prior to phytovolatilisation, the emission rates of materials with a log K_{ow} between 1.1 and 3.6 were calculated for different scenarios using the transport model. Materials with a log K_{OW} between 1 and 3 ought to be effectively taken up by the plant and potentially emitted. However, if the sorption of these materials on the organic soil components is taken into account, the calculated emission rate is drastically reduced and the optimum K_{ow} range shifts in favour of less hydrophilic and sufficiently volatile materials. These model calculations show that the sorption of potentially volatile materials on organic soil components (including the roots) can have a severely limiting effect on phytovolatilisation. The transfer factor (TSCF) and the volatility (K_{AW}) of the materials then play a secondary role. According to the simulation results, methyl tertiary butyl ether with a log K_{OW} of approx. 1 and a log K_{AW} of approx. -2 is emitted more effectively by the plant compared to trichloroethylene and benzene. Hong and coworkers report a pilot project on the remediation of an MTBE-polluted site by planting poplars in Texas (Hong et al. 2001). The indirect emission of MTBE by poplars has already

³ Average value in urban air, RIPPEN 1999 (chapter 'Benzene')

been demonstrated in the laboratory. The extent to which MTBE emission was increased in relation to the unplanted area in the pilot project remains to be ascertained. Nevertheless, the usually high partition coefficients between leaf and atmosphere and the metabolisation of organic materials taken up from air indicate that the net emission of low-volatile, lipophilic compounds from a contaminated area ought to be reduced by planting.

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