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Occurrence of dissolved and particle-bound taste and odor compounds in Swiss lake waters

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ABSTRACT

The occurrence of algal taste and odor (T&O) compounds was investigated in three Swiss lakes which exhibit different nutrient levels from eutrophic to oligotrophic (Lake Greifensee, Lake Zurich and Lake Lucerne). Apart from dissolved T&O compounds, the study also encompassed particle-bound compounds, i.e., compounds that can be released from damaged algal cells during drinking water treatment. A combined instrumental (SPME–GC–MS) and sensory method was applied that allowed to detect and quantify T&O compounds in natural waters in the sub ppt to low ppt-range.

In addition to the prominent T&O compounds geosmin and 2-methyl-isoborneol (MIB), four other T&O compounds could be detected in the lake waters, though all at relatively low concentrations (maximum concentrations of geosmin 19 $\mathrm{ng\,L^{-1}}$, MIB 3 $\mathrm{ng\,L^{-1}}$, β-ionone 27 $\mathrm{ng\,L^{-1}}$, β-cyclocitral 7 $\mathrm{ng\,L^{-1}}$, 2-isobutyl-3-methoxypyrazine 2 $\mathrm{ng\,L^{-1}}$, 2-isopropyl-3-methoxypyrazine 16 $\mathrm{ng\,L^{-1}}$). The concentration peaks typically occurred in the epilimnion during summer concurrent with a high phytoplankton biomass. Consistently, the concentration levels for most of the compounds varied substantially between the three lakes and generally decreased in the order eutrophic Lake Greifensee > mesotrophic Lake Zurich > oligotrophic Lake Lucerne. Furthermore, our data revealed that the occurrence of β-ionone was largely influenced by *Planktothrix rubescens*. This is the first time that a correlation between β-ionone and this cyanobacterium has been reported for natural waters.

1. Introduction

Surface waters are an important source for drinking water throughout the world. Some of the major cities in the world depend to a large extent on reservoirs, natural lakes or river bank filtration to obtain their drinking water. Normally, surface water has to be treated in a multi-step procedure consisting of particle separation, oxidation and adsorption steps to fulfill the requirements with respect to microbiological quality, toxic compounds and aesthetic aspects such as color or taste and odor (T&O). Despite this extensive water treatment, many water utilities are confronted with T&O complaints (Suffet et al., 1996a). On the one hand, this is due to the very low odor thresholds of some T&O compounds (low ppt-range). On the other hand, drinking water treatment is often insufficient with respect to T&O removal. The resulting

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negative perception of the drinking water quality by the public can lead to a lack of trust in the water industry. Therefore, the water utilities are anxious to quickly and efficiently mitigate T&O problems.

Most of the T&O compounds which lead to consumer complaints are formed in surface waters as metabolic products of microorganisms (Suffet et al., 1996b, 1999). Generally, there are two possibilities for water utilities to handle this kind of T&O compounds. As a first option, the production of T&O compounds can be controlled at the source. This means that the growth of phytoplankton in surface waters has to be limited. In the long run, this goal can be achieved by the management of water bodies, i.e., the reduction of the nutrient load into surface waters. As a short-term measure, copper-containing algicides such as copper sulfate have been applied successfully (McGuire et al., 1984; Murray-Gulde et al., 2002). However, while it is feasible to control algae in smaller reservoirs, it is difficult to control (micro)biological growth in larger reservoirs, where tons of algicides are needed to affect algal blooms. Furthermore, the application of algicides often involves negative side-effects, including the potential release of intracellular T&O compounds and cyanotoxins as well as negative effects on the aquatic life. In addition, the accumulation of copper in the sediments might become a long-term problem, with a possible release of copper due to changes in the water chemistry (Haughey et al., 2000). As a second option, drinking water treatment could be optimized with respect to T&O removal. However, an enhanced removal of T&O compounds can only be achieved to an economically feasible degree if the T&O compounds, their origins and their seasonal fluctuations are known. Therefore, it is important to screen surface waters for any compound that could result in a bad taste or odor. In this context, special attention has to be paid to the analysis of dissolved as well as particle-bound T&O compounds. The latter can be an important source of T&O problems when particle-bound compounds are released from damaged algal cells during drinking water treatment.

Previous studies have mainly focussed on the production of T&O compounds by isolated phytoplankton species (Cotsaris et al., 1995; Juttner, 1983; Slater and Blok, 1983b; Watson, 2003) or on the natural occurrence of selected T&O compounds (Durrer et al., 1999; Westerhoff et al., 2005). With a more general approach, it was attempted in an earlier study to comprehensively assess the volatile organic compounds (VOCs) in a eutrophic water body with GC–MS detection and correlate them with phytoplankton species (Juttner et al., 1986). However, this procedure does not allow a specific screening for T&O compounds that could be relevant to drinking water utilities. On the one hand, many VOCs do not change the organoleptic quality of the water because their concentrations are much lower than their respective odor threshold concentrations. On the other hand, potent T&O compounds, which are often present at trace levels, are likely to be missed in the analysis when their weak signals are overlapped by the background of the matrix.

Therefore, an alternative approach which combines classical GC–MS detection with a simultaneous detection at an olfactory detector port (ODP) can be promising. This approach has already found wide application in the food and flavor industry (Campo et al., 2006; Gassenmeier et al., 2001; Zellner et al., 2007), where concentrations of flavor compounds are usually much higher than in drinking water. For this purpose, GC–olfactometry (a review on GC–olfactometry was recently published by Delahunty et al., 2006) coupled to a physical GC detector such as FID or MS is normally used. The literature contains also several descriptions of this combined approach for T&O analysis in drinking waters (Benanou et al., 2003; Bruchet, 2006; Hochereau and Bruchet, 2004; Khiari et al., 1997; Young and Suffet, 1999).

The goal of this study was to evaluate the dissolved and particle-bound T&O compounds in three Swiss lakes with different nutrient levels from eutrophic to oligotrophic (Lake Greifensee, Lake Zurich, Lake Lucerne) by a combined instrumental (SPME–GC–MS) and sensory method. Table 1 summarizes some characteristics of the investigated lakes.

2. Materials and methods

2.1. *Chemicals and water*

T&O compounds which were used as external and internal standards in the quantitative analysis were obtained from Sigma–Aldrich (Buchs, Switzerland) in the highest purity available. Stock solutions (from 10 to 100 $\mu g\,L^{-1}$) were prepared in MilliQ-water. Sodium chloride, which was applied to enhance the extraction of the T&O compounds (see below) was reagent grade and used without further purification.

2.2. *Sampling and sample preparation*

The sampling dates for the different lakes are given in Table 1. A first sampling campaign of Swiss lake waters was performed in 2006 (March–September). The water samples (1 L) were taken from each the epilimnion, the metalimnion and the hypolimnion at fixed depths. In addition, vertical temperature profiles in all lakes and phytoplankton data in Lake Zurich were recorded. For the latter, phytoplankton species were determined and counted according to Utermöhl (1958) under a light microscope (Axiovert 10, Zeiss, Germany). The samples were filtered immediately upon arrival (0.45 μm cellulose nitrate filters, Whatman Schleicher & Schuell, Germany) and divided into a dissolved and a particle-bound fraction. The filtrate with the dissolved fraction was transferred to 100 mL glass bottles. The filter residue with the particle-bound fraction was re-suspended in 1 mL nanopure water and transferred to a 1.5 mL micro tube (Sarstedt, Germany), to which approximately 200 μL glass beads (0.11 mm, B. Braun Biotech International, Germany) were added. Bead beating (Nandakumar and Marten, 2002) with a cell disrupter (FastPrep FP120, Savant Instruments, Holbrook (NY), USA) was applied to break the algal cells. This procedure was run during 6 × 45 s with intermittent cooling of the samples on ice to avoid thermal decomposition of the T&O compounds. A microscopical check of a test sample proved that the cells were destroyed after this preparation step. After sample preparation, all the samples were stored in the dark at 4 °C.

For the analysis of the dissolved fraction, 6 mL of the filtered samples were transferred into 10 mL head-space vials sealed with silicone–PTFE–septa (BGB Analytics, Switzerland).