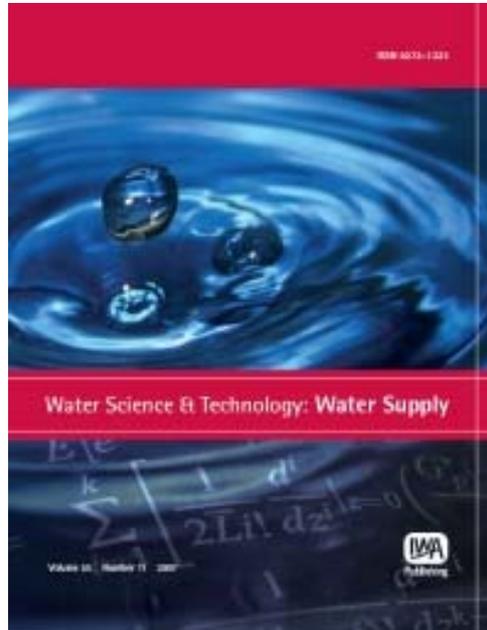


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Pilot scale testing of advanced oxidation processes for degradation of geosmin and MIB in recirculated aquaculture

M. M. Klausen and O. Grønborg

ABSTRACT

The presence of geosmin and 2-methylisoborneol (MIB) in recirculated aquaculture systems has a significant negative impact on the fish production due to poor flavour quality of produced fish and increased risk of rejection by fish processors. Advanced Oxidation Processes has a high potential for removal of geosmin and MIB in water and in this study UV/H₂O₂ and UV/O₃ has been tested in pilot scale in real aquaculture process water. First order degradations constants were between 0.6 (UV/O₃) and 1.2 (UV/H₂O₂) h⁻¹ for geosmin and 1.3 (UV/O₃)–1.5 (UV/H₂O₂) h⁻¹ for MIB. This corresponded to average half-lives between 34–69 minutes for geosmin and between 28–32 minutes for MIB. These values were one order of magnitude higher than previously reported for degradation of geosmin and MIB in demineralised and tap-water. The slower degradation rates were caused by competitive and inhibitive processes from the water matrix. The influence of the water matrix also caused increased energy consumption with EEO values 16 to 38 times higher than previously reported for geosmin and MIB removal in tap water. Improved feasibility of removing geosmin and MIB in recirculated aquaculture systems by AOPs requires pre-treatment to minimize the impact of the water matrix on the degradation kinetics.

Key words | advanced oxidation, competitive processes, kinetics, taste and odour, water matrix

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INTRODUCTION

In recirculated aquaculture systems, soluble compounds which are not removed by the water treatment plant, accumulates due to the high degree of water recycling. Among these compounds, the taste and odour compounds geosmin and 2-methylisoborneol (MIB) has a significant negative impact on the fish production. Due to their lipophilic properties geosmin and MIB readily and rapidly accumulates in fish flesh thereby resulting in poor flavour quality and subsequent delays in harvesting due to rejection of the cultured fish by processors (Schrader & Rimando 2003). In fact, off-flavours add \$15 to \$23 million annually to catfish production costs in the USA and is a significant problem to aquaculture worldwide (Hanson 2003). Off-flavour problems are likely to increase worldwide due to the

greater demand for aquaculture products by consumers and an increase in recirculated aquaculture production due to eutrophication of water bodies from traditional aquaculture farming.

The accumulation rate of geosmin and MIB in fish flesh has been reported to be exponential and equilibrium is established within 10–15 days (Howgate 2004). Equilibrium concentrations of geosmin and MIB in the fish flesh will be up to 400 (Geosmin) and 200 (MIB) times the concentration in the surrounding water (Howgate 2004). For a typical water concentration of geosmin of 10 ng/l and a standard trout or salmon of around 750 g with 10% fat this means that equilibrium concentrations in the fish will be around 4 µg/kg fish (Howgate 2004). Compared with the

human threshold value for taste of geosmin of around 0.3 µg/kg this stresses the importance of continuously treating the process water in recirculated aquaculture systems to lower the concentration of geosmin and MIB.

Today the recirculated aquaculture industry does not apply dedicated water treatment technologies for removal of taste and odour compounds. Instead, the strategy used by fish farmers to avoid rejection of the expensively produced fish is to transfer the fish to tanks with fresh water supply for one to two weeks to desorb the accumulated geosmin and MIB from the fish flesh (Howgate 2004). This is an expensive and capacity demanding strategy and application of water treatment technology for continuous removal of geosmin and MIB seems as an attractive alternative.

Within drinking water treatment, taste and odour problems due to geosmin and MIB are to a certain extent handled using granular or powdered activated carbon (Hargesheimer & Watson 1996; Crozes *et al.* 1999; Gillogly *et al.* 1999; Bruce *et al.* 2002). However, the concentrations of geosmin and MIB in drinking water is typically well below the levels in aquaculture process water. Furthermore, the higher concentrations of soluble organic matter in general in recirculated aquaculture systems indicates that the use of activated carbon to remove geosmin and MIB does not seem feasible due to the competition for adsorption sites on the carbon and consequently lower lifetime and frequent replacements of the carbon.

Conventional chemical oxidation technologies, like chlorination, potassium permanganate and ozonation, have also been investigated in relation to the removal of geosmin and MIB in drinking water (Lalezary *et al.* 1986; Park *et al.* 2007). These technologies have all shown low removal rates and capacities, probably because these oxidants react selectively with unsaturated and aromatic structures in organic compounds, which are not characteristic for geosmin and MIB that are both saturated cyclic alcohols. In some studies ozone has, however, been far more efficient compared to the other conventional oxidants in removing geosmin and MIB (Glaze *et al.* 1990). The higher efficiency in this study was probably caused by a decay of ozone to the far more powerful oxidant the hydroxyl radical (OH·) under slightly alkaline conditions, thereby transferring the ozonation process to an advanced

oxidation process (AOP). These results have entailed a growing interest in applying advanced oxidation technology (AOT) for removal of geosmin and MIB from water.

Accordingly a number of scientific studies with different AOPs have been conducted all showing a large potential for degradation of MIB and geosmin (Ferguson *et al.* 1990; Koch *et al.* 1992; Lawton *et al.* 2003; Rosenfeldt *et al.* 2005; Bellu *et al.* 2008). However, all these studies has been conducted under laboratory conditions using demineralised or tap-water spiked with geosmin and MIB with no or very low influence from the water matrix on the degradation. One of the major factors governing the efficiency of the AOP technology is the fraction of OH· available for the oxidation of target contaminants compared to the fraction of OH· scavenged by the background water matrix. Free radical scavengers compete for the hydroxyl radicals and make the degradation of organic contaminants slower and less cost-effective. Simultaneously, other compounds may affect the formation rate of the free radicals.

The purpose of this study was therefore to study the degradation rates of geosmin and MIB by two advanced oxidation processes, i.e. UV/H₂O₂ and UV/O₃ in full scale using real process water from a recirculated aquaculture system to get more insight into the influence of the water matrix on the degradation rate and the cost-effectiveness of the AOPs.

MATERIALS AND METHODS

Test site

The test site for the UV/H₂O₂ and UV/O₃ processes was Fyns Laksefisk (In English: Fuens Troutfarm), a small recirculated fish farm producing 700.000 sea trouts (*Salmo trutta*) a year (26 ton). The sea trouts are produced in tanks from which water is continuously drawn for mechanical and biological treatment and oxygenation in a water treatment plant. From the treatment plant the water is returned to the productions tanks. The fish farm operates the production process with 99% recycling of the water entailing a high accumulation rate of colloidal and soluble compounds not removed in the water treatment plant.

Reagents

The process water was spiked with a certified geosmin and MIB standard from Supelco (Sigma Aldrich) with a concentration of 100 µg/ml of geosmin and MIB in methanol.

Residual ozone in water samples was quenched using sodiumsulphite (Na_2SO_3) while residual hydrogen peroxide was removed catalytically by adding 350 µL 5.7 M KI solution to each 20 mL sample for analysis of geosmin and MIB.

Experimental procedure and test equipment

The experiments was conducted as batch degradation experiments around a tank of 0.5 m³ of volume. The tank was filled with real process water taken after the mechanical filtration in the water treatment plant. The water was spiked with geosmin and MIB to secure the presence of the compounds prior to the degradation experiments. The spiking was done using a glass Hamilton syringe and a certified geosmin and MIB standard cooled to -18°C to minimize vaporization of geosmin and MIB during spiking. After the spiking, mixing of the geosmin and MIB was secured by recirculating the process water over the batch container for 2 retention times (0.5 hours) before test start and initial sampling. To minimize vaporization of geosmin and MIB during mixing and testing, the tank was designed with an opening of only 30 cm and in addition the batch container was filled completely with process water to minimize the surface area from which geosmin and MIB could vaporize. These precautions were done based on experience given by Bellu *et al.* (2008) who found that air stripping had a significant impact on volatilization of geosmin during photocatalytic degradation experiments.

The AOP test equipment consisted of the Aqua Chameleon UV/hydrogen peroxide and Aqua Chameleon UV/Ozone system manufactured by Skjølstrup & Grønborg Aps—ULTRAAQUA.

The Aqua Chameleon UV/Hydrogen peroxide system

The Aqua Chameleon UV/hydrogen peroxide system was comprised of a Grundfos DMS-12-3 diaphragm dosing pump with synchronous motor technology with a dosing range between 0.0025 to 12 l/h. The hydrogen peroxide was

dosed directly into the circulation pump for the UV reactor to ensure complete mixing before the inlet to the UV reactor. The UV reactor was an inline stainless steel reactor fitted with a medium pressure UV lamp with a total power output of 3.5 kW and a total UV output in the wavelength range between 200–400 nm of 0.9 kW. The UV reactor was fitted with a UV irradiance sensor for continuous monitoring of UV dose.

The Aqua Chameleon UV/Ozone system

The Aqua Chameleon UV/Ozone system was comprised of a dry air ozone generator model GA10 from PacificOzone-Technology. The generator was operated at maximum power output of the air-cooled ceramic and titanium dual action reactor cell and at airflow of 0.57 Nm³/h. At these conditions the generator supplied 3 g ozone/hour. The ozone was introduced to the process water through a Mazzei venturi injector with a 95% mass transfer efficiency of ozone at the stated conditions. The ozone containing water was afterwards led to a stainless steel UV reactor of 1-meter in length and a diameter of 6.3 cm. The UV lamp consisted of an amalgam lamp emitting light at 254 nm. The UV-amalgam lamp had a total power output of 130 W and a UV-C output of 50 W. After passage of the UV reactor the treated process water was led through a passive cascade stripper for removal of excess ozone which had not been photolysed and from here back to the batch container. Ozone containing off-gas was vented to the out-side during the experiments. A principle drawing of the test set-up is shown in Figure 1.

Experimental conditions

Five repeated batch degradation experiments were conducted with each technology at fixed operational conditions as given by the supplier (Table 1). For each experiment the removal kinetics of geosmin and MIB was determined.

Analytical methods for process water quality characterization

Temperature, pH and conductivity were measured with calibrated probes. Turbidity was measured as optical density

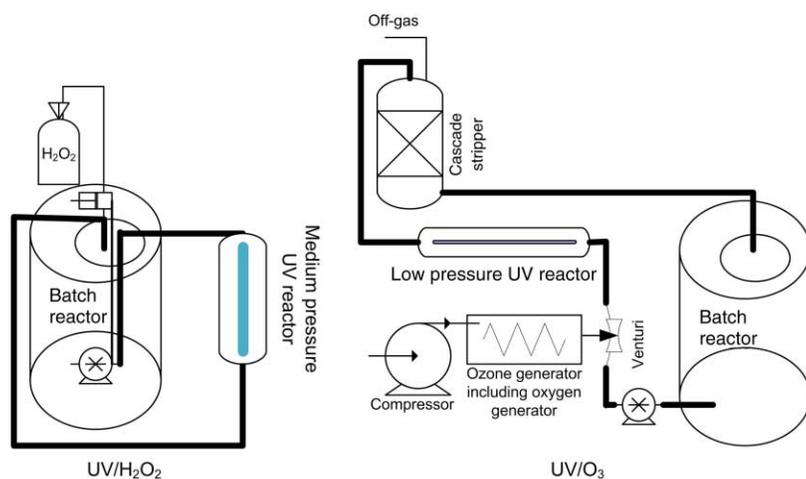


Figure 1 | Experimental set-up of the degradation experiments.

at 650 nm and UV absorbance/Transmission was measured at 254 nm using a Hach-Lange DR 5000 UV/VIS spectrophotometer. Alkalinity and color was measured according to Standard Methods 2320B and 2120C respectively.

Analytical methods for ozone and hydrogen peroxide measurements

During UV/O₃ experiments residual ozone concentrations after the UV reactor was measured by the indigo trisulfonate method, with AccuVac ampoules (Hach-Lange, www.hach-lange.com).

During UV/H₂O₂ experiments residual hydrogen peroxide concentrations after the UV reactor was measured using a colorimetric method where hydrogen peroxide reacted with potassium titanium oxalate in acid solution to form the yellow pertitanic acid complex measured spectrophotometrically at 400 nm. The procedure consisted of adding 1 ml sample to a cuvette followed by addition of 0.200 ml reagent A (50 g/l potassium titanium oxalate) and 0.200 ml reagent B (1:18 diluted sulphuric acid $d = 1.84$) followed by absorbance measurements at 400 nm using

a Hach-Lange DR 5000 UV/VIS spectrophotometer. Concentrations were calculated based on a standard curve.

Analytical methods for geosmin and 2-MIB

Geosmin and 2-MIB were analyzed using head-space solid-phase micro extraction (HS-SPME) coupled to a GC/MS system as suggested by [Watson *et al.* \(2000\)](#). The SPME fibre coating used for extracting geosmin and MIB was Polydimethylsiloxane/Divinylbenzene (PDMS/DVB). Samples were collected in 20 mL dark vials containing 5 g NaCl. The vials were filled completely with no headspace. Before analysis, the samples were split into two samples of 10 mL each in 20 mL dark vials. The compounds were adsorbed to the PDMS/DVB fiber in the headspace of the vials upon 30 minutes of heating at 60 °C under shaking conditions. Desorption is made in the injection port at 250 °C for 5 minutes. The compounds are analyzed by the GC/MS. Geosmin is analyzed using the m/z 122 and the MIB is analyzed by the m/z 95. The limit of detection for the method is around 1 ng/l for geosmin and MIB respectively.

Table 1 | Operational conditions for the UV/H₂O₂ and UV/O₃ technologies

Parameter	Unit	UV/H ₂ O ₂	UV/O ₃
Flow	(m ³ /h)	2	0.3
UV-dose (medium pressure/low pressure)	(mJ/cm ²)	600	500
H ₂ O ₂ /O ₃ -dose	(mmol/l; mg/l)	0.5; 17	0.2; 10

RESULTS AND DISCUSSION

Water matrix composition

A couple of months prior to the degradation experiments a one week sampling and analysis campaign including several water quality parameters with influence on the OH-radical

Table 2 | Process water quality at Fyns Laksefisk

Parameter	Unit	Average	Range (n = 5)
UV ₂₅₄ absorbance	cm ⁻¹	0.5	0.54–0.56
UV ₂₅₄ transmittance	%T (1 cm)	28.3	27.6–28.8
Turbidity (Abs 650 nm)	cm ⁻¹	0.004	0.003–0.005
Color	mg Pt/l	41	37–47
pH	–	7.1	7.0–7.2
Conductivity	μS/cm	3,400	N/A
Alkalinity	mg HCO ₃ ⁻ /l	281	N/A
Geosmin	ng/l	12.4	6–21
MIB	ng/l	20.8	8–40

formation as well as OH-radical scavenging were conducted to characterize the variability in the water quality of the fish farm. Samples were taken each day for five consecutive days to assess the variability in the basic water quality as well as in the content of geosmin and MIB. Table 2 summarizes the results of the analysis campaign at Fyns Laksefisk.

The process water at Fyns Laksefisk showed high absorption of UV-light with UV-254 absorbance (UV-254 transmittance) values ranging from 0.54 to 0.56 cm⁻¹ (27.6–28.8%). The high UV absorbance of the process water corresponded well to the high color content ranging from 37 to 47 mgPt/l. Due to the high color content and UV absorbance the applied UV dose for the batch degradation experiments were adjusted based on calculations of minimum required UV dose at the wall of the reactor taking into account the molar absorption coefficients of hydrogen peroxide ($\epsilon_{200-300\text{nm}} = (140 - 1) \text{ M}^{-1} \text{ cm}^{-1}$) and ozone ($\epsilon_{254\text{nm}} = 3,300 \text{ M}^{-1} \text{ cm}^{-1}$). Turbidity measured as optical density as absorbance at 650 nm showed very low turbidity values between 0.003 and 0.005 cm⁻¹. The pH of the process water was around neutral. The process water had relatively high conductivity and alkalinity with measured values around 3,400 μS/cm and 281 mg HCO₃⁻/l respectively. The high conductivity was primarily caused by the high recirculation rate in the system combined with addition of sodium chloride. Both bicarbonate and chloride ions are known radical scavengers with high reaction rate constants with hydroxyl radicals ($k_{\text{OH},\text{HCO}_3} = 8.5 \times 10^6 \text{ M}^{-1} \text{ s}^{-1}$; $k_{\text{OH},\text{Cl}} = 3 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$) (Liao *et al.* 2001) so the presence of these ions in high

concentration may significantly influence the degradation of geosmin and MIB in real aquaculture process water. The measured concentrations of geosmin and MIB were in the range of 6–21 ng/l and 8–40 ng/l respectively showing that the content of the taste and odour compounds varied significantly from day to day. Due to this it was decided to spike the process water before the degradation experiments to ensure that geosmin and MIB were present.

Oxidation of geosmin and MIB by UV/H₂O₂ and UV/O₃

Degradation kinetics

The Aqua Chameleon UV/H₂O₂ and UV/O₃ systems were applied for oxidation of geosmin and MIB in the process water of the recirculated aquaculture farm. Five repeated experiments for each technology were conducted. Initial geosmin concentrations were within 4.2 to 6.7 ng/l while the initial MIB concentrations were within 32 to 87 ng/l for all the degradation runs. The degradation data revealed 1st order removal during the experiments for both geosmin and MIB with correlation coefficients for the 1st order fit (R^2) between 0.8 and 1. The degradation data from all the experiments with the two technologies are shown in Figures 2 and 3. Due to the variations in the initial concentrations of geosmin and MIB the concentrations are displayed as relative concentrations for comparison of the individual runs.

The degradation results for the five repeated experiments showed similar degradation kinetics for each technology thus verifying the reproducibility of the controlled experiments. In all experiments the degradation rate for both geosmin and MIB was faster for the UV/H₂O₂ process. The actually obtained first order degradations constants for geosmin and MIB for the UV/H₂O₂ process was between $1.2 \pm 0.3 \text{ h}^{-1}$ for geosmin and $1.5 \pm 0.3 \text{ h}^{-1}$ for MIB. This corresponded to average half-lives of 34 and 28 minutes for geosmin and MIB, respectively. For the UV/O₃ process the first order rate constants were $0.6 \pm 0.3 \text{ h}^{-1}$ for geosmin and $1.3 \pm 0.2 \text{ h}^{-1}$ for MIB and these corresponded to half-lives of 69 and 32 minutes respectively. Compared to previously reported half-lives of 3.5 minutes ($k = 11.9 \text{ h}^{-1}$) and 8 minutes ($k = 5 \text{ h}^{-1}$) of geosmin and MIB in controlled laboratory photocatalytic

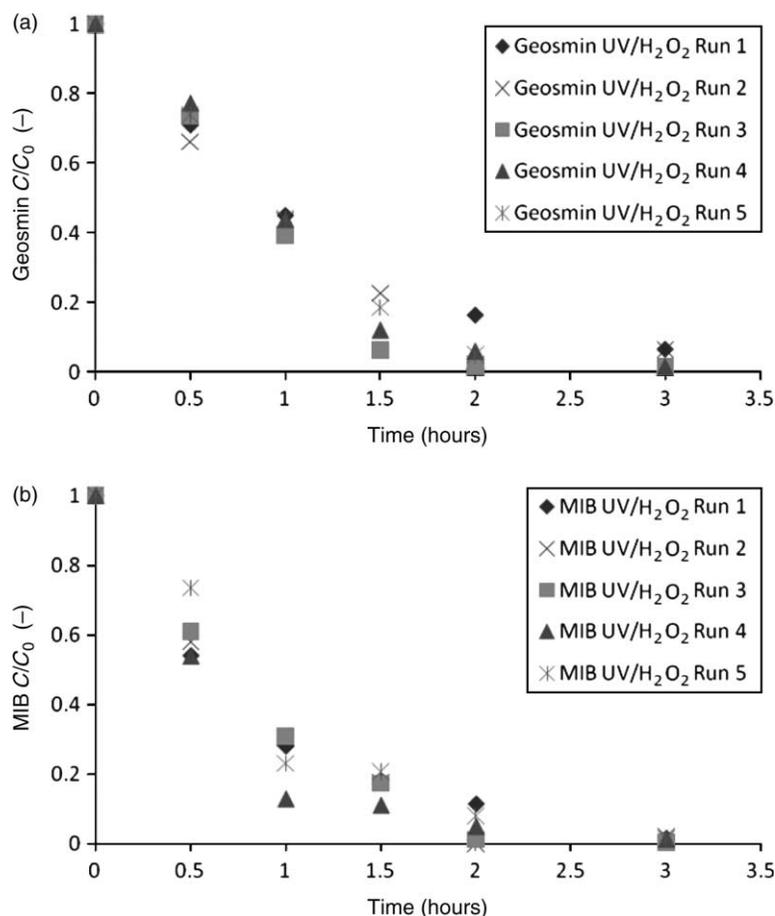


Figure 2 | Pilot scale degradation of geosmin and MIB compounds by UV/H₂O₂; (a) Geosmin, (b) MIB. Initial concentrations of geosmin were between 6.1–6.5 ng/l and of MIB between 32–37 ng/l.

AOP experiments in demineralised water (Lawton *et al.* 2003) this showed the significant impact of the water matrix on the degradation rate in real aquaculture process water. During the experiments the UV absorbance at 254 nm declined with around 40% and the color content of the process water declined with up to 60% showing that competitive oxidation processes to geosmin and MIB oxidation were taking place acting as a major contributor to the slow degradation rates. In addition, other competitive oxidation processes like the inorganic radical scavengers was most probably also acting during the experiments. The extent of these could however not be distinguished from the results. The removal of UV absorbing and color causing compounds was mostly pronounced during UV/O₃ experiments. This indicated that part of the ozone reacted selectively as molecular ozone with aromatic structures of

the organic matter giving rise to the color of the process water. This would lead to less dissolved ozone available for photolysis into hydroxyl radicals and consequently to a lower degradation rate of geosmin and MIB. No accumulation of dissolved ozone was observed during the experiments. During the UV/H₂O₂ experiments, hydrogen peroxide accumulated slightly in the process water showing that the photolysis of hydrogen peroxide was incomplete and affected by the high UV absorption. This showed the sensitivity to high UV absorption of the UV/H₂O₂ process due to the low molar absorption coefficient of H₂O₂ in the wavelength window of the medium pressure lamp ($\epsilon_{200-300\text{ nm}} (140 - 1) \text{ M}^{-1} \text{ cm}^{-1}$). Despite the impact of the UV absorbance on the photolysis of hydrogen peroxide still a higher formation of hydroxyl radical occurred leading to higher degradation rates compared with the UV/O₃

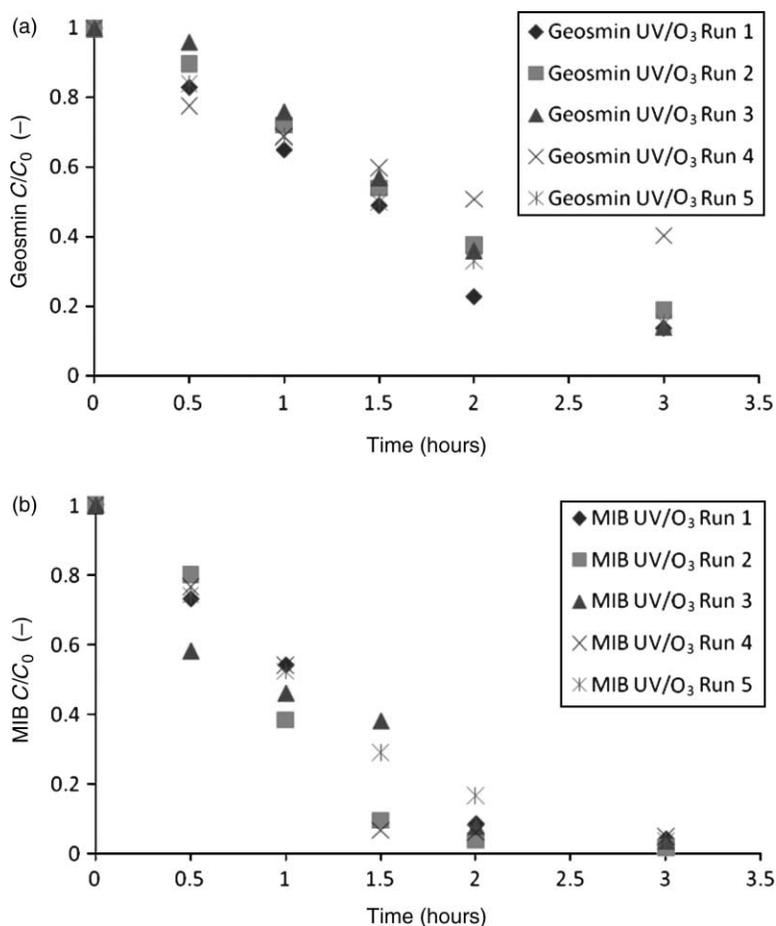


Figure 3 | Pilot scale degradation of geosmin and MIB compounds by UV/O₃; (a) Geosmin, (b) MIB. Initial concentrations of geosmin were between 4.2–6.7 ng/l and of MIB between 57–89 ng/l.

process. The pH also declined during the degradation runs showing that the oxidation of the organic matter was not complete and leading to formation of weak acid groups like e.g. carboxylic acids. The turbidity remained low and unchanged during the experiments and did not influence the degradation.

Energy consumption aspects

The far slower degradation rate in real aquaculture process water observed in this study compared to previously reported degradation rates from laboratory experiments also impacted the cost effectiveness of the AOP processes tested. The energy consumption of the AOP processes can be calculated by the Electrical energy required (in kilowatt hours) per order of magnitude of contaminant removal in

1 cubic meter of water (EEO) (Bolton *et al.* 2001). The calculated EEO values were 16 and 13 kWh/m³ for geosmin and MIB for the UV/H₂O₂ process and 19 and 8 kWh/m³ for the UV/O₃ process respectively. This is significantly higher than EEO values reported by Rosenfeldt *et al.* 2005 who reported EEO values of 0.5–2 kWh/m³ for geosmin and MIB removal in raw and treated surface water which was found fairly feasible. Based on the findings in the present study, application of the tested AOP process for geosmin and MIB removal in recirculated aquaculture systems does not seem feasible without pre-treatment like activated carbon filtration to remove macromolecular organic compounds to minimize the impact of the water matrix on the degradation kinetics and thus reducing the total oxidant demand for removal of geosmin and MIB. In addition, it should be noted that the actual increase in

energy consumption for a recirculated aquaculture farm might be lower than indicated above due to the possibility of integrating the AOP processes into the existing treatment system using the oxygenation cone for ozone addition and existing UV reactors for photolysis however this integration possibility needs further investigations.

CONCLUSION

Advanced oxidation processes has a great potential for degrading geosmin and MIB in water. Being the first study to publish results from a pilot scale test on AOP for removal of off flavors in real process water of a recirculated fish farm, the results of this study show that the complex water matrix significantly influences the degradation rate and cost effectiveness of the UV/H₂O₂ and UV/O₃ processes tested. The pilot scale degradation experiments revealed first order degradation constants and half-lives one order of magnitude higher than reported for degradation of geosmin and MIB in demineralised and tap-water. The slower degradation rates was caused by competitive oxidation processes with other organic matter and inorganic radical scavengers as well as by inhibition of radical formation in the UV/H₂O₂ process due to a high UV-absorption of the process water. The competitive and inhibitive processes also caused an increased energy consumption as calculated by the EEO factor yielding values 16 to 38 times higher than previously reported for removal of geosmin and MIB in tap water. To improve the feasibility of removing geosmin and MIB in recirculated aquaculture systems by Advanced Oxidation Processes it is necessary to combine the AOP with pre-treatment to minimize the impact of the water matrix on the degradation kinetics. Furthermore increased feasibility could be gained through integration of the AOP processes into the existing water treatment system of a recirculated aquaculture farm, however this needs further research.

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