

Removal of geosmin and 2-methylisoborneol during managed aquifer recharge: batch and column studies

S. K. Maeng, C. D. T. Abel, S. K. Sharma, N. S. Park and G. L. Amy

ABSTRACT

Managed aquifer recharge is a robust barrier in the multi-barrier approach to supply safe drinking water. The removal performance of geosmin and 2-methylisoborneol through managed aquifer recharge was investigated using batch and column experiments. Batch experiments were carried out to investigate the removal of geosmin and 2-methylisoborneol (MIB) in the presence of different types of biodegradable organic matter using different types of water. Five different types of water spiked with 70–293 ng/L of geosmin and MIB were used in batch reactors, and complete removal of geosmin and MIB (down to the detection limit) was achieved in all cases. Soil column studies showed that biodegradation contributed to the removal of geosmin and MIB by 23 and 31%, respectively (empty bed contact time: 17 hours). The removal of geosmin and MIB appeared to be influenced more by microbial activity than the initial concentrations of geosmin and MIB. Adsorption was found to be the dominant mechanism (major role) followed by biodegradation (minor role) for geosmin and MIB removals during soil passage. Managed aquifer recharge can therefore be used as a robust barrier to remove taste and odor (T&O) causing compounds.

Key words | 2-methylisoborneol, adenosine triphosphate, biodegradable organic matter, geosmin, managed aquifer recharge, riverbank filtration

S. K. Maeng (corresponding author)
Department of Civil and Environmental
Engineering,
Sejong University, 98 Gunja-Dong,
Gwangjin-Gu, Seoul 143-747,
South Korea
E-mail: smaeng@sejong.ac.kr

C. D. T. Abel
S. K. Sharma
G. L. Amy
UNESCO-IHE Institute for Water Education,
P.O. Box 3015, 2601 DA Delft,
The Netherlands

N. S. Park
Water Research Center,
K-Water Institute,
462-1 Jeonmin-dong, Yuseong-Gu,
Daejeon, South Korea,
305-730

G. L. Amy
Water Desalination and Reuse Center,
King Abdullah University of Science and
Technology (KAUST), Thuwal,
Kingdom of Saudi Arabia

INTRODUCTION

Geosmin and 2-methylisoborneol (MIB) are odorous metabolites formed by cyanobacteria and actinomycetes and impart a musty/earthy taste and odor in drinking water (Izaguirre *et al.* 1982; Jelen *et al.* 2003). Due to their very low odor threshold concentrations, geosmin and MIB are easily detected at concentrations as low as 5–10 ng/L (Lin *et al.* 2002; Ho *et al.* 2007). Traditional water treatment processes (coagulation, flocculation, sedimentation and filtration) are not effective in removing geosmin and MIB dissolved in raw water (Bruce *et al.* 2002). Reiss *et al.* (2006) demonstrated that microfiltration or ultrafiltration membrane systems were not efficient at removing geosmin and MIB (5–40% removal). Advanced oxidation (e.g. ozonation) and powdered activated carbon (PAC) processes have been effective for geosmin and MIB removal; however, the presence of natural organic matter (NOM) affects the removal efficiencies of geosmin and MIB, increasing

energy use and requiring a high PAC dose for water utilities (Ho *et al.* 2002; McDowall *et al.* 2009). The effective removal of these compounds, especially in the presence of NOM characteristics, is therefore a growing concern for water utilities.

Managed aquifer recharge (MAR) is often considered as a cost-effective treatment process providing safe drinking water for towns and small communities (Dillon 2005). MAR (e.g. riverbank filtration, artificial recharge and soil aquifer treatment), a soil/aquifer-based natural treatment process, can potentially function as a robust barrier for the elimination of organic micropollutants including geosmin and MIB. MAR is capable of removing many different types of contaminants including pharmaceutically active compounds, endocrine disrupting compounds and pesticides (Ray 2008; Maeng *et al.* 2011a, 2011c). During soil passage, many contaminants are attenuated through

biodegradation, sorption and mixing with natural groundwater (Maeng *et al.* 2010, 2011b). A soil passage experiment using column studies, which simulates MAR systems, significantly reduced biopolymers (e.g. polysaccharides, protein-like substances that are readily biodegradable fractions in bulk organic matter) (Maeng *et al.* 2008). A risk assessment conducted by Page *et al.* (2010) demonstrated that the residual risks associated with organic chemicals were low based on their intensive monitoring studies carried out from the Parafield aquifer storage transfer and recovery site in South Australia.

Terauchi *et al.* (1995) reduced the required PAC concentrations after biofiltration to about 1/3 to remove MIB below 15 ng/L. A number of previous studies have reported on the removal of odor compounds by MAR (i.e. riverbank filtration) (Chorus *et al.* 1992; Jüttner 1995). Geosmin was reduced by 95% from a monitoring well located 50 m from the bank, and riverbank filtration can remove not only geosmin but also other fragrance compounds (Jüttner 1995). To date, no research has been carried out on the contribution of biodegradation to the removal of geosmin and MIB by differentiating between biodegradation and adsorption mechanism during soil passage, and the role of microbial activity, which can be determined by adenosine triphosphate (ATP), in the removal of geosmin and MIB has not been investigated. The objective of this study was to investigate the removal of geosmin and MIB in the presence of different types of biodegradable organic matter using different types of water. Additional objectives were to determine

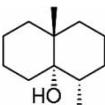
the role of microbial activity, determined by ATP, in the removal of MIB and geosmin during MAR using batch and column experiments. The change in geosmin and MIB concentrations during sample storage at 4 °C with and without 0.45 µm filtration was also investigated.

MATERIALS AND METHODS

Chemicals/source water

Geosmin and MIB standard samples were purchased from Sigma-Aldrich at a concentration of 100 µg/mL in methanol. Using Milli-Q water (Advantage A10, Millipore), stock solutions of 100 µg/L were prepared. Working solutions in different sources of water were prepared every day from the stock solution to minimize the biodegradation of geosmin and MIB for soil column studies. Table 1 shows physical–chemical properties of geosmin and MIB. Biodegradable synthetic organic matter (BSOM) based on the formation of organic matter originating from ozonation was selected to investigate the effect of organic carbon by-products (assimilable organic carbon) on the removal of geosmin and MIB during MAR. BSOM contained aldehyde compounds (formaldehyde 200 µg/L and glyoxal 60 µg/L) and carboxylic compounds (sodium acetate 800 µg/L and sodium formate 600 µg/L) (Urfer & Huck 2001), and was used for batch studies as additional carbon sources. A water supply company in the Netherlands, DUNEA, has

Table 1 | Physical–chemical properties of geosmin and MIB (US EPA 2009)

Characteristics	Geosmin	MIB
Structure		
CAS	019700-21-1	002371-42-8
Molecular weight	182.31	168.28
Log K_{ow}	3.57	3.31
Henry's law constant ^a	6.66×10^{-5}	5.76×10^{-5}
Solubility (mg/L)	294.9	345.4
BIOWIN ^b	2.37 (weeks–months)	2.19 (days–weeks)

^aPirbazari *et al.* (1992).

^bUltimate survey model (US EPA 2009).

investigated the feasibility of an advanced oxidation process as a pre-treatment process for MAR (Lekkerkerker *et al.* 2009). This may increase assimilable organic carbon, which may enhance the removal of organic micropollutants by co-metabolism during soil passage. Therefore, synthetic forms of organic matter were added in the batch study to investigate whether or not additional carbon sources may influence the removal of geosmin and MIB.

Geosmin and MIB analysis

MIB and geosmin were measured using solid phase microextraction and gas chromatography (Thermo Trace Ultra GC Ultra)-mass spectrometry (Thermo Trace Ultra DSQ). Ten milliliters of standards or samples was added to a 20 mL screw-capped vial with stirring followed by the internal standard (d_3 -MIB at 50 ng/L). The fiber was placed in the injector of the gas chromatography (GC)-mass spectrometry (MS) after 30 min of headspace extraction was conducted at 70 °C. The temperature program of the GC column (Optima 5 Accent, 0.25 μ m, 28 m, Macherey-Nagel) began in the following order (3 min: 40 °C, 2 min: up to 165 °C by 5 °C min⁻¹, 3 min: up to 250 °C by 30 °C min⁻¹). $m/z = 171$ and $m/z = 168$ were used for selecting the ion monitoring (SIM) mode of MIB and the internal standard of d_3 -MIB, respectively. The detection limits of geosmin and MIB were determined as 3.4 and 4.4 ng/L, respectively. A detailed description of this analysis was documented by Kutschera *et al.* (2009).

DOC, SUVA and ATP

All samples were stored at 4 °C after 0.45 μ m filtration to prevent the biodegradation of organic matter. Also, unfiltered samples stored at 4 °C were analyzed for geosmin and MIB to compare with filtered samples with 0.45 μ m. The concentration of bulk organic matter was determined as dissolved organic carbon (DOC) by a total organic carbon analyzer (TOC-VCPN Shimadzu). UV absorbance was measured at 254 nm by a UV/Vis spectrophotometer (UV-2501PC Shimadzu), and specific UV absorbance (SUVA) was calculated by dividing the UV254 absorbance with DOC concentration. In this study, biodegradable organic carbon (BDOC) was defined by the DOC change instead of by

measuring assimilable organic carbon (AOC). Both the BDOC and AOC methods have drawbacks, and AOC was not measured in this study. Adenosine triphosphate (ATP) was measured to determine the microbial activity in soil columns. Wet sand samples between 1 and 2 g collected from batch and column studies were suspended in autoclaved tap water (50 mL). High-energy sonication at 40-W power was used to detach ATP associated with sand (Branson W-250D Sonifier with a D^{1/4} 5-mm microtip). ATP concentration in the suspension was measured. A single 2-minute sonication treatment was adequate to obtain more than 90% of the attached biomass represented as ATP (data not shown). A detailed description of the methods and materials used for the ATP extraction procedure is given in Magic-Knezev & van der Kooij (2004).

Experimental setups

Batch studies

Sand in batch reactors was previously acclimated with the Meuse River water for 30 days. Batch experimental set ups (1 L glass bottles) using 200 g of acclimated silica sand (grain size 0.8–1.25 mm) were used to assess the effects of biodegradable organic matter on the removal of geosmin and MIB using different types of water, simulating a long residence time (30 days in an orbital shaker set at 100 rpm) during MAR. All batch reactors were not closed in order to maintain the reactors under oxic conditions. Six batch reactors in triplicate with a working volume of 800 mL were prepared. The six different types of water used are: (1) Meuse river water, The Netherlands (MR), (2) Meuse river water spiked with BSOM (formaldehyde 200 μ g/L, glyoxal 60 μ g/L, sodium acetate 800 μ g/L, and sodium formate 600 μ g/L) as an additional carbon source, (3) secondary effluent from a wastewater treatment plant in Hoek van Holland, The Netherlands (SE), (4) water from an experimental container used for cultivation of the common reed, *Phragmites australis* (CR), and (5) non-chlorinated tap water (NCTW), Delft, The Netherlands (AOC close to 10 μ g/L). For the CR, *Phragmites australis* was excavated from the shore of a lake located in Delft, The Netherlands and (trans)planted in a specially-constructed container. The water level of the container

was maintained at the container surface. The plants were incubated under constant temperature (16 °C) with a 14-h light period. CR was collected from the valve located at the bottom of the container. The intent of using CR for batch reactors was to simulate water with high contents of humic substances. CR exhibited high aromaticity from SUVA analysis, and CR contained organic matter which originated from soil organic matter. (6) The Meuse River without sand was also prepared, and samples were taken intermittently to determine their biodegradation rates in the MR.

Column studies

Acclimated silica sand was used for a 300 mm long double-jacketed column with a 50 mm inner diameter (XK50/30, Amersham Pharmacia Biotech, Sweden). The inner part of the columns was packed with acclimated sand, while the outer part was used to control the prevailing ambient temperature by flowing water from a chiller. The samples were kept in a dark room to minimize any potential effect of photodegradation, and the empty bed contact time (EBCT) was 17 hours. Three sand columns (SC1, SC2 and SC3) were deployed to assess the role of microbial activity in the removal of geosmin and MIB with a differentiation between biodegradation and adsorption. SC1 and SC2 were acclimated with MR containing geosmin and MIB for 2 months. Similar to the case of batch experiments mentioned above, the acclimation process was continued until both columns stabilized with respect to DOC reduction. After the acclimation period, NCTW and MR served as the feed to the SC1 and the SC2, respectively. The SC1

aimed at an assessment of biodegradation of geosmin and MIB under low microbial conditions using NCTW (i.e. low biodegradable organic matter). In contrast to the acclimation period of 60 days for SC1 and SC2, SC3 had no acclimation period and received demineralized water (DW) containing sodium azide (NaN_3 , 20 mM) to maintain the column under abiotic conditions.

RESULTS AND DISCUSSION

Effect of background biodegradable organic matter on geosmin and MIB removal

Laboratory-scale batch experiments were conducted to analyze the effect of biodegradable organic matter present on the removal of geosmin and MIB. Biodegradable organic carbon ($\text{BDOC}_{30\text{-day}}$) was defined by the DOC change during 30 days of incubation (Table 2). SE exhibited the highest $\text{BDOC}_{30\text{-day}}$ value of 6.0 mg/L followed by CR (4.1 mg/L), MR+ BSOM (2.1 mg/L), MR (1.0 mg/L), and NCTW (below limit of quantification). SUVA values of 3.3 L/mg-m for CR were followed by SE (3.1 L/mg-m), MR (2.7 L/mg-m), and MR + BSOM (1.8 L/mg-m), indicating the aromaticity of organic matter in each sample (Table 2). The low SUVA value observed in MR + BSOM (2.4 L/mg-m) was attributed to the aliphatic nature of BSOM added to the MR. Each reactor had a different degree of aromaticity in organic matter and $\text{BDOC}_{30\text{-day}}$; however, the complete removal (not detected) of geosmin and MIB was observed for all batch reactors (Table 2). In this study, there was no significant impact of biodegradability of organic matter on the

Table 2 | Geosmin and MIB concentrations for batch reactors

	Geosmin (ng/L) 0-day	Geosmin (ng/L) 30-day	MIB (ng/L) 0-day	MIB (ng/L) 30-day	BDOC (mg/L) 30-day	SUVA (L/mg-m)
MR	210 (11) ^c	N.D. ^a	255 (2)	N.D.	1.0	2.7
MR + SOM	235 (3)	N.D.	263 (8)	N.D.	1.9	2.0
SE	71 (2)	N.D.	235 (6)	N.D.	5.5	3.1
CR	279 (2)	N.D.	291 (3)	N.D.	4.1	3.3
NCTW	226 (3)	N.D.	273 (7)	N.D.	B.L.Q. ^b	–

^aN.D.: not detected (the limits of detection for geosmin and MIB were determined as 3.4 and 4.4 ng/L, respectively).

^bB.L.Q.: below limit of quantification.

^c(): standard deviation.

removal of geosmin and MIB. This may be due to the long residence time (30 days) of batch reactors, or to the fact that sorption was a dominant mechanism for geosmin and MIB.

Geosmin and MIB were partially removed in the reactor without sand, and were 22 and 92 ng/L at 21-day (Figure 1(a)), respectively. Complete removal (not

detected) of geosmin and MIB would have been possible with bioacclimated sand. Figure 1(a) shows that MIB was more difficult to remove compared to geosmin. The BIOWIN model was used to estimate the biodegradability of geosmin and MIB (US EPA 2009). According to BIOWIN 3, geosmin also degrades slightly faster than

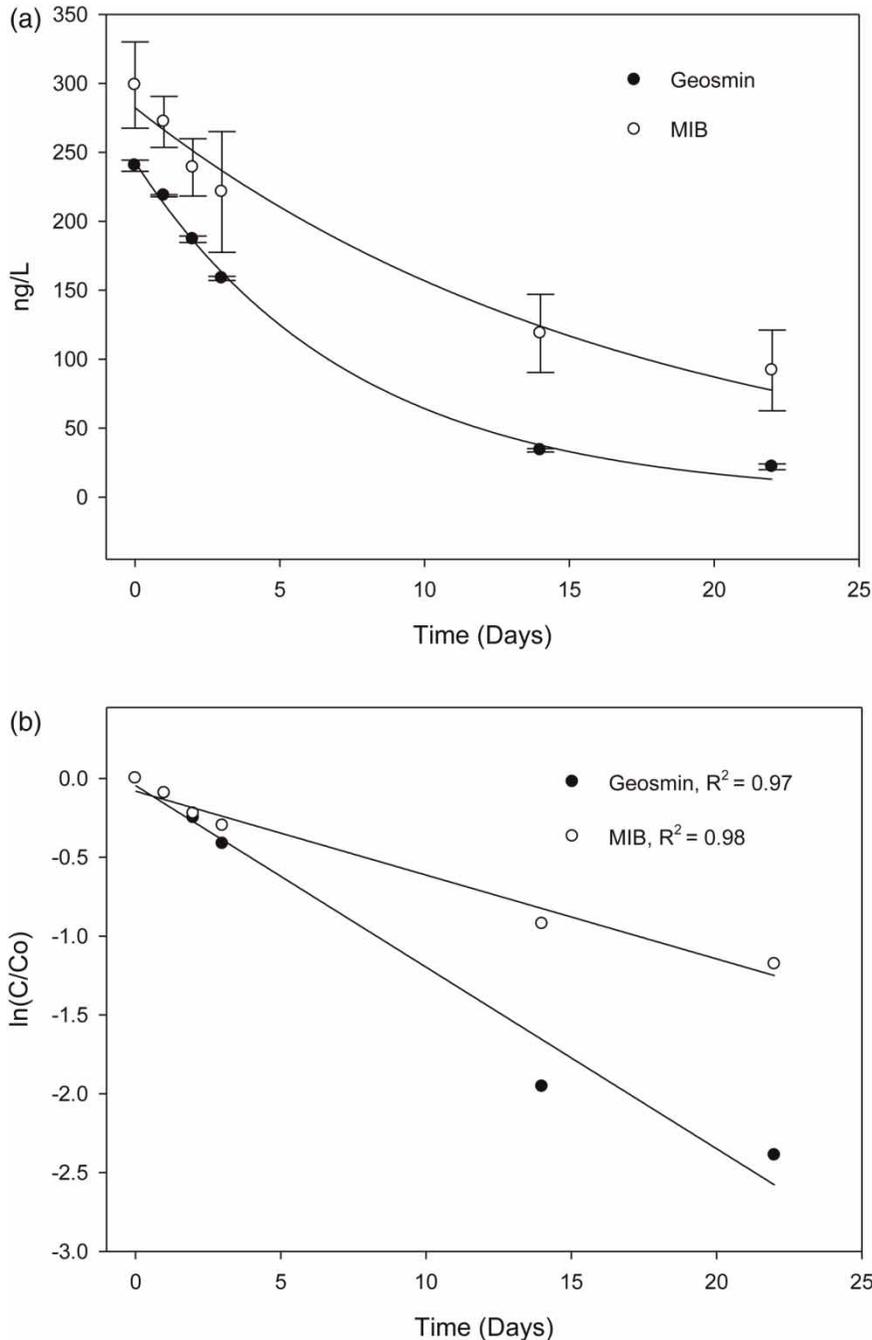


Figure 1 | (a) Removal of geosmin and MIB in the MR in batch reactor (no acclimated sand); (b) pseudo-first-order kinetic plots for geosmin ($k = 0.115 \text{ d}^{-1}$) and MIB ($k = 0.053 \text{ d}^{-1}$).

MIB (Table 1). Pirbazari *et al.* (1992) and Ho *et al.* (2002, 2004) suggested that the more volatile nature of geosmin (i.e. Henry law constants) and its lower solubility contributed to its higher removal (Table 1). Samples were also taken intermittently during the 30 days of the batch experiment, and the removal of geosmin and MIB showed a pseudo-first-order reaction. Reaction rate coefficients were 0.115 and 0.053 d⁻¹ for geosmin and MIB, respectively (Figure 1(b)). This result was consistent with that of Ho *et al.* (2007) who found that the biodegradation of geosmin and MIB in natural water followed pseudo-first-order kinetics, and the reaction rate coefficients of geosmin and MIB were 0.064 and 0.075 d⁻¹, respectively. Anderozzi *et al.* (2006) and Ho *et al.* (2007) suggested the aerobic biodegradation of organic compounds as a secondary substrate (i.e. geosmin and MIB), which is significantly lower than the primary substrate (i.e. NOM) in natural water, and is considered as a pseudo-first-order reaction according to the Michaelis–Menten equation. In this study, the reaction rate coefficient found for geosmin was faster compared to that in the previous study carried out by Ho *et al.* (2007).

As a control experiment, the degradability of geosmin and MIB in samples stored at 4 °C was investigated using samples with and without 0.45 µm filtration. Even for samples maintained at 4 °C (24-h), geosmin in samples without 0.45 µm filtration were reduced by 30% compared to those samples filtered with 0.45 µm (Figure 2). However, MIB was only slightly changed between filtered and unfiltered samples. 0.45 µm filtration is a necessary step to maintain geosmin and MIB concentrations during storage at 4 °C.

Role of microbial activity in geosmin and MIB removal

Laboratory-scale soil column studies were conducted to analyze the role of microbial activity in the removal of geosmin and MIB during soil passage. Previously, SC1 and SC2 had an acclimation period of 60 days with the MR. In order to reduce microbial activity in SC1, the feed water was changed from MR to NCTW after the acclimation period. The SC2 column was prepared in the same way as SC1, except that the feed water was not changed to NCTW from MR. The DOC in NCTW ranged between 1.8 and 2.0 mg/L and

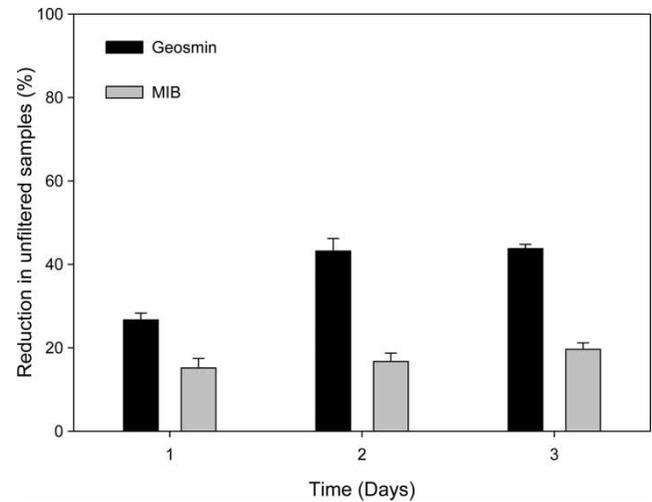


Figure 2 | Reduction of geosmin and MIB concentrations in unfiltered samples (the Meuse River) stored at 4 °C.

consisted of a very low biodegradable fraction. SC1 is aimed at the assessment of the biodegradation of geosmin and MIB at low microbial activity using low BDOC water (i.e. NCTW). In contrast to SC1 and SC2, SC3 was packed with sand whereby no acclimation period was provided, and it was fed with demineralized water containing sodium azide (20 mM) to suppress microbial activity in the column in order to determine the removal of geosmin and MIB by sorption. ATP concentrations associated with sand for SC1, SC2 and SC3 were 30, 102 and 0.6 ng ATP/cm³ of dry sand, respectively (Maeng *et al.* 2011b). No data are available on the removal of ATP concentrations in a real MAR system. However, Magic-Knezev and van der Kooij have reported ATP concentrations associated with sand from a slow sand filter, which is a similar concept to bank filtration but with a shorter travel time), ranging from 18 to 93 ng ATP/cm³ of dry sand (Magic-Knezev & van der Kooij 2004). Moreover, ATP concentrations in filtrates from SC1, SC2 and SC3 were 23.5, 781, 3.2 ng ATP/L, respectively. Thus, ATP concentrations associated with sand, and in filtrate, reflected the microbial activities in each column. Low microbial activity in SC1 by introducing NCTW decreased the ATP concentrations associated with sand by 30%. However, geosmin and MIB were still significantly removed in SC1 and SC2 (>97%) (Figure 3). SC3 resulted in removal of geosmin and MIB of 75 and 67%, respectively, in SC3. Based on this result, biodegradation

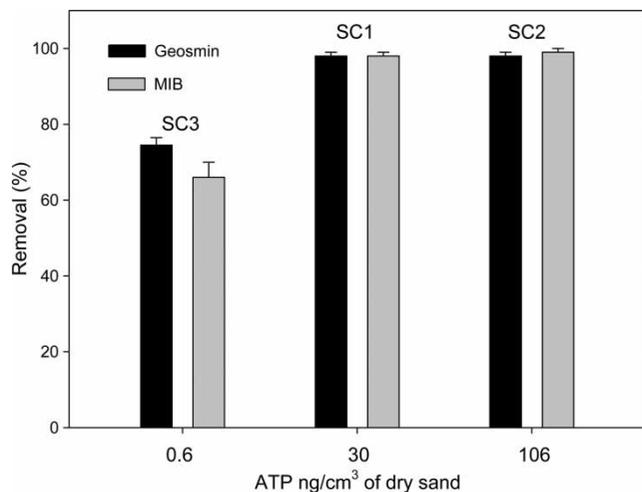


Figure 3 | Removal of geosmin and MIB at different microbial activities determined by ATP.

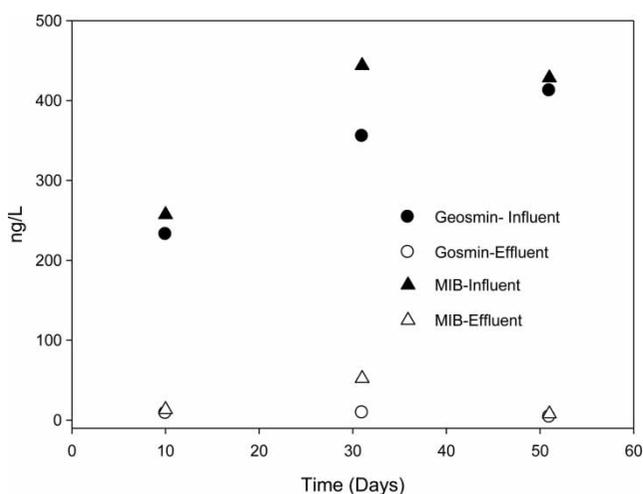


Figure 4 | Effect of initial concentrations on geosmin and MIB removal during soil passage (EBCT: 11 hours).

enhanced the removal of geosmin and MIB by 23 and 31% in soil column studies, respectively. Moreover, different acclimation periods changed the removal of geosmin and MIB during soil passage; further study is therefore required to determine whether microbial diversity was influenced by acclimation periods.

From this study, adsorption was found to be the dominant mechanism (major role) followed by biodegradation (minor role) for geosmin and MIB removals during soil passage. This is likely due to their moderate high octanol-water partition coefficient values which reflect their hydrophobic

characteristics (geosmin $\log K_{ow}$: 3.57 and MIB $\log K_{ow}$: 3.31) (Table 1).

Figure 4 shows that the removal was not significantly changed by increasing the concentration of geosmin and MIB from 200 to 400 ng/L. This implies that the removal of geosmin and MIB seems to be influenced more by microbial activity rather than by the initial concentrations of geosmin and MIB.

CONCLUSIONS

This study examined the removal of geosmin and MIB under different sample matrices and the role of microbial activity during soil passage. The different bulk organic matter characteristics present in different types of water tested in this study did not influence the removal of geosmin and MIB. In general, geosmin was easier and faster to remove compared to MIB. The removal of geosmin and MIB showed a pseudo-first-order reaction, and the reaction rate coefficients of geosmin and MIB were 0.115 and 0.053 d^{-1} , respectively. Column studies demonstrated that geosmin and MIB were significantly removed under biotic conditions (>97%). The results obtained from columns with a different ATP concentration showed that biodegradation contributed to the removal of geosmin and MIB removal by 23 and 31%, respectively. Based on the results of this study, it can be concluded that MAR can potentially function as a robust barrier for the elimination of taste and odor causing compounds (i.e. geosmin and MIB) from water.

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