



## Evaluation of the short-term fate and transport of chemicals of emerging concern during soil-aquifer treatment using select transformation products as intrinsic redox-sensitive tracers



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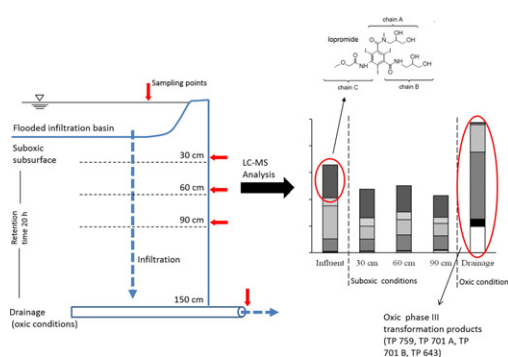
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### HIGHLIGHTS

- Two wet-dry cycles of a full-scale infiltration basin were monitored.
- Known products from oxic transformation were identified to serve as intrinsic redox tracers.
- The occurrence of intermediates confirmed limited availability of dissolved oxygen.

### GRAPHICAL ABSTRACT



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### ABSTRACT

In this study, known products from oxic transformation of the X-ray contrast medium iopromide were introduced for the first time as intrinsic tracer for in situ characterization of the transition zone between oxic and suboxic conditions during the initial phase of soil-aquifer treatment (SAT). Two wet-dry cycles of a full-scale infiltration basin were monitored to characterize hydraulic retention times, redox conditions, removal of bulk organic parameters and the fate of chemicals of emerging concern (CECs). Tracer tests at the site showed an average hydraulic retention time of <20 h before collection in drainage pipes located approximately 1.5 m below surface. Dissolved oxygen at different depth rapidly depleted and only increased towards the end of the flooding event. Transformation of iopromide and all known intermediates to persistent transformation products (TPs) usually occurring during oxic biodegradation was very limited in samples from suction cups immediately underneath the basin. But transformation was complete in samples collected from the drainage outlet indicating that dissolved oxygen had been introduced to the system before sample collection in the combined drainage outlet. Similar to iopromide and its TPs, removal of several CECs (diclofenac, bezafibrate, mecoprop, TCEP) was inefficient after 90 cm infiltration (<35%) but significantly enhanced in the combined drainage outlet (>80%). These results highlight that the analysis of iopromide along with its intermediates and persistent TPs can serve as a promising probing tool to determine overall efficiency of CEC biodegradation and to identify potential in situ oxygen limitations.

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## 1. Introduction

Water scarcity is a growing challenge in the world and freshwater resources increasingly run short to satisfy demand. Due to the uneven distribution of water resources and increasing degree of urbanization, water shortages are often a climate-related regional problem. Many communities depend on water resources that are partially replenished intentionally or unintentionally with treated wastewater (Pal et al. 2010). The presence of chemicals of anthropogenic origin in these water resources, such as pharmaceuticals, personal care products, household chemicals, endocrine disrupting compounds, flame retardants, plasticizers and pesticides including their respective metabolites and transformation products (TPs) has been recognized for a longer time (La Farre et al. 2008; Celiz et al. 2009; de Jongh et al. 2012). Even though most of these so-called 'chemicals of emerging concern' (CECs) are present at a low concentrations (nanogram per liter or even pikogram per liter, ng/L or pg/L range), there are concerns regarding potential adverse human and environmental health effects (Jones et al. 2004; Schwarzenbach et al. 2006; Du et al. 2014). Therefore, understanding the fate and transport of these chemicals, an assessment of potential adverse health effects of CECs, as well as more effective treatment systems for their mitigation are challenges that need to be addressed.

Soil-aquifer treatment (SAT) is a rather inexpensive, sustainable treatment and reclamation technology to generate higher-quality water from treated domestic wastewater effluents for non-potable and potable uses (Regnery et al. 2013). SAT consists of a controlled passage of treated wastewater through porous media mainly for purification purposes, as well as for seasonal and multiannual storage. SAT includes a three-component treatment process consisting of the infiltration zone, vadose zone, and the underlying aquifer. Purification processes in SAT systems include filtration, biotransformation, mineralization, physical adsorption, chemical precipitation, and ion-exchange (Kanarek and Michail 1996; Maeng et al. 2011; Regnery et al. 2013). While this natural treatment system is highly attractive due to its low carbon footprint and the minimal generation of residuals, drawbacks of this technology are the rather large physical footprint requirements and a lack of understanding of system dynamics resulting in variable qualities of the recovered water (Regnery et al. 2013).

SAT proved to be effective in the removal of turbidity, total nitrogen, phosphorus, pathogenic bacteria, protozoa, and viruses (Gerba et al. 1991; Idelovitch et al., 2003). A significant removal of dissolved organic carbon (DOC) after SAT has been reported (Quanrud et al. 2003; Drewes et al. 2006; Amy and Drewes 2007). Previous studies have also demonstrated that SAT is effective in reducing the concentrations of various CECs that are still present in wastewater treatment plant (WWTP) effluents (Quanrud et al. 2003; Amy and Drewes 2007; Ternes et al. 2007; Laws et al. 2011). The attenuation of trace organic chemicals during SAT strongly depends on structural moieties of the chemical, hydrogeological conditions, residence times, travel distances, redox conditions, temperature, availability of a primary substrate, microbial activity and functionality (Grünheid et al. 2008; Maeng et al. 2011; Li et al. 2014; Alidina et al. 2014). However, some residual trace organic chemicals, such as X-ray contrast media, antibiotics, chlorinated flame retardants and antiepileptic drugs (e.g., carbamazepine, primidone), persist during SAT and can occur in reclaimed water and recovered groundwater samples in the elevated ng/L-range (Snyder et al. 2003; Regnery et al. 2013).

Several studies investigated the removal efficiency of organic matter and CECs during SAT. However, current knowledge is insufficient to properly reveal the transformation dynamics of CECs as a function of predominant redox conditions in particular during the initial phase of infiltration. The availability of dissolved oxygen plays a key role in determining the mobility, dissolution, transformation and toxicity of most CECs (Regnery et al. 2015a). Already at low dissolved oxygen

concentrations (defined as suboxic conditions with  $DO < 1$  mg/L and little denitrification with  $\Delta NO_3^- < 0.5$  mg N/L) the microbial degradation of redox-sensitive CECs can be abated (Regnery et al. 2015a). While anoxic conditions can be distinguished by monitoring different redox parameters ( $NH_4^+/NO_3^-$ ;  $Mn^{2+}/Mn^{4+}$ ;  $Fe^{2+}/Fe^{3+}$ ;  $SO_4^{2-}/H_2S$ ), in situ characterization of the transition between oxic and suboxic conditions is often hindered by inappropriate sampling techniques (e.g., suction cup samplers, redox probes).

During this study, we utilized for the first time known transformation products (TPs) of the X-ray contrast medium iopromide as intrinsic tracers of oxygen limitations. This probing tool might be suitable to characterize the biotransformation of other CECs during short-term SAT. In order to test this hypothesis, this study investigated the performance of a full-scale SAT facility with short hydraulic retention time during travel through a 1.5 m deep vadose zone.

## 2. Materials and methods

### 2.1. Study site description

In this study, an infiltration basin operated by the Abwasserverband Braunschweig, Germany and located in a wetland area of approximately 150 ha was selected for the field experiments. The WWTP Steinhof, with an annual treatment capacity of approximately 22 million  $m^3$ , is employing full biological nutrient removal (i.e., nitrogen and phosphorus) by operating a three stage activated sludge system (with anaerobic, anoxic and oxic zones). About one third of the secondary effluent flow is fed into the wetland area. The entire wetland area is equipped with a drainage system that dates back to the end of the 20th century but still catches about 85% of the infiltrated water. The recovered water is subsequently drained via open ditches to the Aue-Oker-Canal and finally discharged into the river Oker. Within this area, infiltration basin S62 (Fig. S1, Supplemental information) with a surface area of approximately 1225  $m^2$  was selected for full-scale SAT experiments. Water applied to the infiltration basin and percolating through the subsurface is collected in the historic drainage pipes located approximately 1.5 m below surface. The drainage pipes for this infiltration basin are combined into a common discharge point located approximately 150 m to the northwest of the basin. The combined drainage also collects water from adjacent infiltration basins, which were not operated during the entire period of the experiments.

### 2.2. Basin operation and sampling procedures

Prior to the experiments, the infiltration basin was flooded on a regular basis every two to three weeks for a period of approximately 3 months. Two experiments were executed in consecutive trials (September and November). Before the field trials, the ground surface of the infiltration basin was mown and plowed to reduce potential uptake of water by plants growing in the basins. Precise flow measurement devices at the delivery point were not available, but the influent volume was estimated based on basin dimensions (length and width) and the impounding depth. The empty infiltration basin was flooded once to a water level of 36 cm above ground surface (resulting in a recharge volume of approximately 440  $m^3$ ) in September and 40 cm (resulting in a recharge volume of approximately 490  $m^3$ ) in November 2015.

Samples were taken from secondary effluent (basin influent), from the flooded infiltration basin (2 samples/day taken at opposite side of delivery point), and from the combined drainage outlet (after vadose zone treatment). For the second experiment (November) additional suction cups (UMS, Germany) and oxygen optodes (PreSens, Germany) were installed at 30 cm, 60 cm and 90 cm depth beneath the infiltration basin.

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