

**ENA OF HETEROCYCLIC HYDROCARBONS BY ADDING HYDROGEN PEROXIDE IN GROUND-
WATER CIRCULATION WELLS
- A FIELD-BASED STUDY ON A LARGE PHYSICAL MODEL SCALE -**

Oliver Trötschler, Thomas Haslwimmer, Anne Sagner*, Andreas Tiehm*, Hans-Peter Koschitzky

*Technologiezentrum Wasser, Karlsruher Straße 84, D-76139 Karlsruhe, Germany
Phone: +49 721-9678 220, Fax: +49 0721-9678 101
E-mail: tiehm@tzw.de

VEGAS, Institut für Wasserbau, Universität Stuttgart, Pfaffenwaldring 61, D-70550 Stuttgart, Germany
Phone: +49 711 685 7021, Fax: +49 711 685 7020
E-mail: oliver.troetschler@iws.uni-stuttgart.de

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1 Introduction

Heterocyclic Hydrocarbons (NSO-HET) are ingredients of tar oil, commonly found downgradient of former gasworks sites. Typical NSO-HET are benzofurans, methyl-benzofurans, methylquinoline, acridine or carbazole. During investigations of MNA (monitored natural attenuation) remediation strategies, it was found that most NSO-HET are highly mobile due to their high water solubility and low biodegradation rates. In addition, some were found to be highly toxic and carcinogenic (LOH & YU, 2000, BUNDY ET AL., 2001, ROBBIANO ET AL., 2004, SEYMOUR ET AL., 1997). In particular under anaerobic conditions, NSO-HET biodegradation rates are low. However, aerobic biological degradation was found to be effective.

Based on the extension and contaminant distribution of the plume (~ 800 m long) downgradient of a former gasworks "Testfeld Süd" (TFS) in Southern Germany, the most applicable technology for enhancing the natural degradation of PAH, BTEX and NSO-HET was selected and tested under controlled conditions in a large physical model (Large Flume of VEGAS). The investigations focused on a technology for a homogeneous infiltration of electron acceptor solutions such as oxygen and hydrogen peroxide to provide the bacteria with molecular oxygen. An initial infiltration of oxygen (air-saturated water) during the adaptation of microorganism to aerobic biodegradation was followed by a time-limited addition of hydrogen peroxide to achieve an oxygen concentration up to 23 mg/L in the model aquifer. An almost complete degradation of NSO-HET was found.

On the basis of numerical simulations and lab experiments, it was found that natural dispersion will not lead to a wide-ranging homogeneous distribution and mixing of the oxygen in the aquifer. The Groundwater Circulation Wells technology (GCW) can be applied to achieve a maximum mixing of the electron acceptor solution with the groundwater. A spherical groundwater circulation is induced by means of ex- and infiltration ports in vertical wells. Infiltration and exfiltration ports are located in hydraulically separated filter sections (MOHRLOK ET AL., 2000).

Field monitoring at TFS showed the degradation of all PAH along the plume, except for acenaphthene. The NSO-HET are still present in the plume due to anaerobic conditions.

In a second step, the technology is to be applied in a field trial of about one year's duration.

2 ENA experiment "Large Flume" of VEGAS

The technology of GCW and its effectiveness for creating a homogeneous distribution of oxygen was tested in a large physical model: the Large Flume of VEGAS. A numerical model was developed, calibrated and verified by fluorescent tracer tests in the Large Flume in order to describe the flow and mixing processes of the GCW. The numerical approach was then used for the planning of the GCW's field application.

The Large Flume (Fig. 1) is 7.5 m long, 1 m wide and 3 m high. It is packed to a depth of 2.5 m with quaternary coarse sand (1 – 2 mm grain size, layered filling). The hydraulic conductivity of 4.5×10^{-3} m/s is comparable to the field site (EBERHARDT ET AL., 2002). A constant groundwater flow was maintained, using constant flux inflow (12 l/h) and constant head outflow conditions. The mean travel rate of the anaerobic, degassed groundwater in the flume was approximately 0.4 m/d.

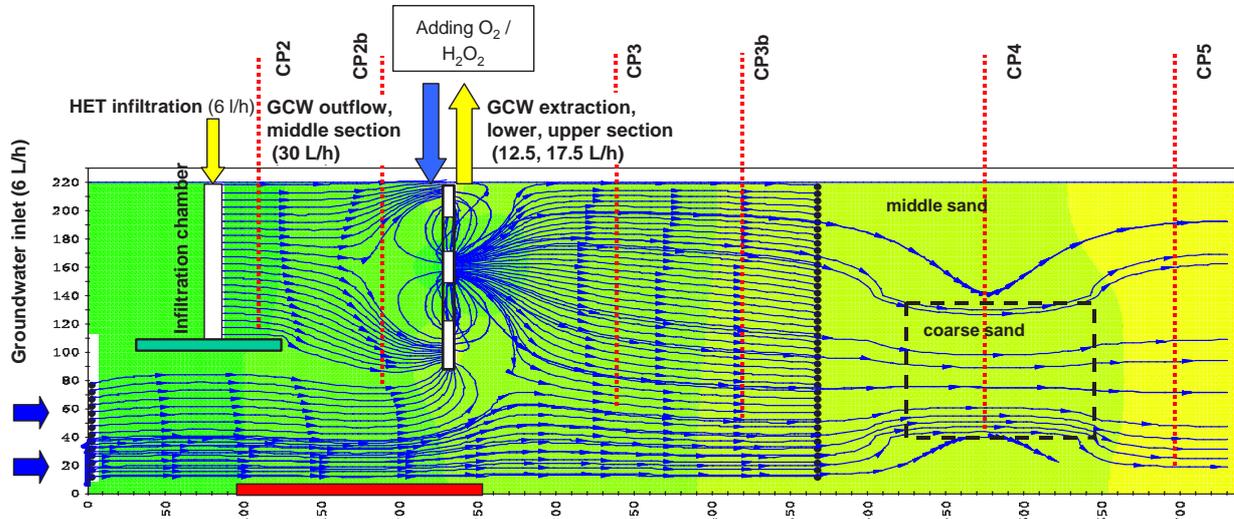
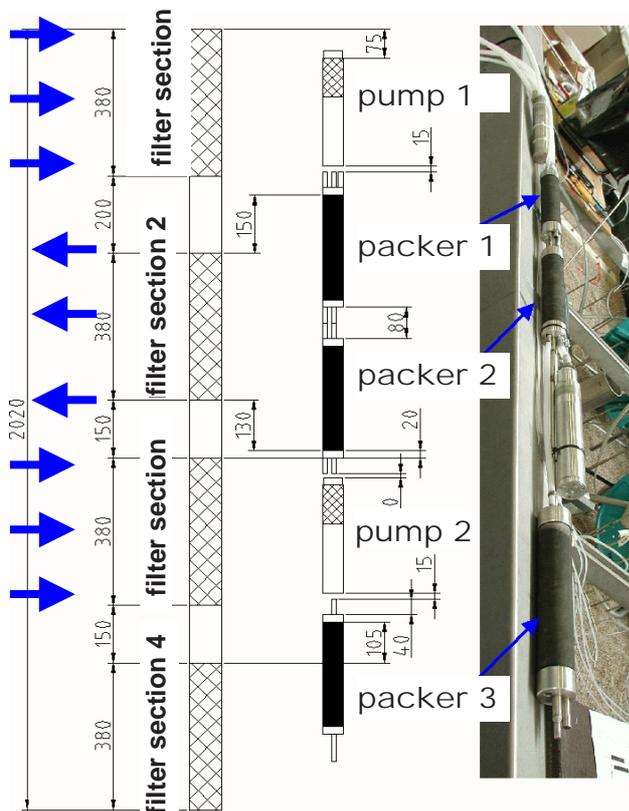


Figure 1: Hydraulic scheme of the Large Flume showing the location of GCW and control planes (CP2, 2b, 3, 3b, 4 und 5), measured in cm

A representative mixture of NSO-HET was constantly infiltrated into the model aquifer by a screened infiltration chamber covering the entire width of the model aquifer to create a contaminant plume. The mixture contained benzofuran, 2-methyl-benzofuran, 2, 3-dimethylbenzofuran, 6-methyl-quinoline, dibenzothiophene, acridine and, as an example of PAH that is poorly degradable, acenaphtene. The initial concentrations of the single NSO-HET ranged between 20 and 100 $\mu\text{g/L}$, comparable to the NSO-HET concentration of the field site.



One GCW (3"- version) was installed 1.6 m downgradient of the contaminant source zone. Plastic submersible pumps (1") were operated to extract the groundwater via two separated filter chambers of 30 cm length located in the upper and lower section of the GCW (Fig.2). Oxygen (air) or hydrogen peroxide was added and the water was re-infiltrated through the screened middle section of the GCW. The ratio between groundwater flux and discharge of the GCW was 2.5. The spherical groundwater circulation captured approximately 80% of the entire groundwater, hence leading to a complete capture of the contaminant plume.

Figure 2: Construction of GCW in 3"- size, measured in mm

The aerobic biological degradation of NSO-HET was controlled by balancing the contaminant mass passing the control planes. In total, four control planes were installed, each equipped with 9 sampling points and one additional well for pumping-based integrative sampling. The mass flux of NSO-HET at each control plane was calculated by multiplying the concentration at the single sampling ports by the numerically calculated groundwater discharge passing the extrapolated representative cross-section area that was influenced during sampling (100 mL each).

After a 42-day period of aeration the mixing of hydrogen peroxide started, aiming at a concentration of 50 mg/L H_2O_2 in the GCW. During the aeration period the aerobic biodegradation was initialised downgradient of the GCW. After six weeks of hydrogen peroxide release, an almost complete contaminant degradation was observed. NSO-HET concentration dropped below 0.5 $\mu\text{g/L}$ downgradient of the GCW.

2 Hydraulic and numerical investigations

The aim of the numerical simulation was to describe the complex hydraulic system of a GCW in order to design the field application. A numerical model using MODFLOW 2000 and MT3D (GMS 4.0, Brigham Young University, Provo Utah, USA, <http://emrl.byu.edu>) was developed on the basis of a large physical model (Large Flume).

The hydraulic investigations were carried out to prove the hydraulic mixing efficiency of GCW technology and to determine the hydraulic permeability distribution, average dispersion coefficient (α_t : 0.14 cm, $\alpha_t / \alpha_l = 0.1$), mean travel rate (0.41 m/d), anisotropy (2.6), effective porosity (0.27) in the physical model in order to calibrate the numerical model.

A numerical model grid (size: 2.5 x 2.5 x 2.5 cm) was established to describe the physical structures of the model aquifer: homogeneous middle sand (7.5 x 2.5 x 1 m) packed in layers, surrounding one coarse sand block (2.5 x 1 x 1 m). The location and discharge of the GCW were defined by numerical simulations to ensure a complete capturing of the contaminated groundwater.

The hydraulic mixing efficiency of the GCW was tested during the first fluorescent tracer test that lasted for 21 days. Tracer (uranine, 700 $\mu\text{g/L}$) was continuously added to the groundwater leaving the infiltration chamber for 14 days.

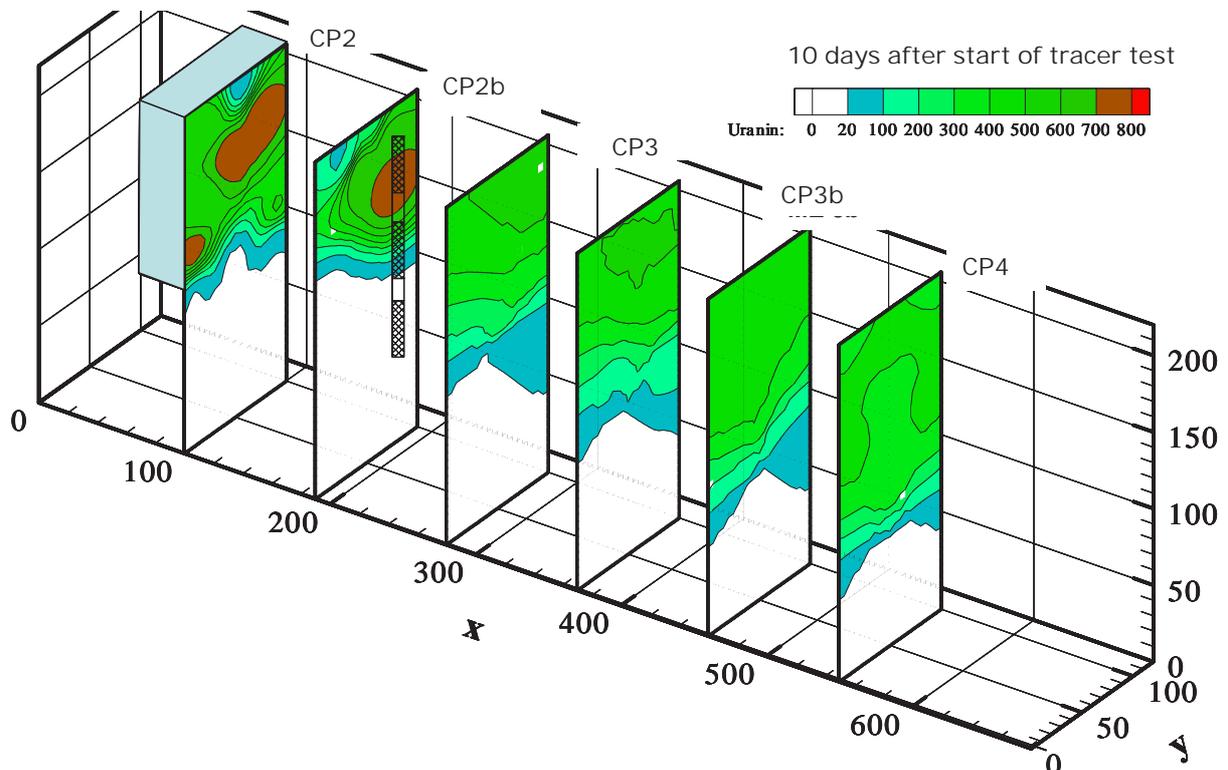


Figure 3: Distribution of fluorescent tracer (uranine, $\mu\text{g/L}$), ten days after start of infiltration

Ten days after tracer start, samples were taken at all 45 ports. A rather inhomogeneous distribution of fluorescent tracer close to the infiltration chamber (CP2, Fig. 3) and upgradient of the GCW (CP2b) was observed. Downgradient of the GCW (CP3, 3b, and CP4) and within the GCW, an almost homogeneous tracer distribution was observed. This confirmed the mixing properties of the GCW technology. Fiber optical systems and online fluorescence analyzers (Hermes Messtechnik, <http://www.hermes.de>) were used to plot tracer breakthrough curves at six sampling ports. Numerically calculated values of the mean tracer breakthrough time differed by $\pm 10\%$ from the plotted experimental data.

A second tracer test was started for a finer calibration of the numerical model. Uranine (4 mg/L, three days) was continuously added to the mixing chamber of GCW. The number of sampled ports for online fluorescence measurement was doubled and the model structure was changed from a homogeneous distribution of hydraulic permeability to a block structure that is characterized by layers of different permeability from 10- 30 cm thickness. During inverse numerical simulations, the permeability of the block zones was varied between 2.6 and 8×10^{-3} m/s, a permeability of 4.5×10^{-3} m/s was allotted to more than 70 % of all cells.

A direct comparison (Fig. 4) of simulated and measured data confirmed the successful calibration and validation of the numerical approach. The results of the numerical simulation ensure the safe design of the GCW and were used to determine the mass flow of NSO-HET passing the control planes. The proposed proceeding (tracer test to calibrate numerical model) is the basis for a safe design and for the hydraulic optimization of the planned pilot GCW application.

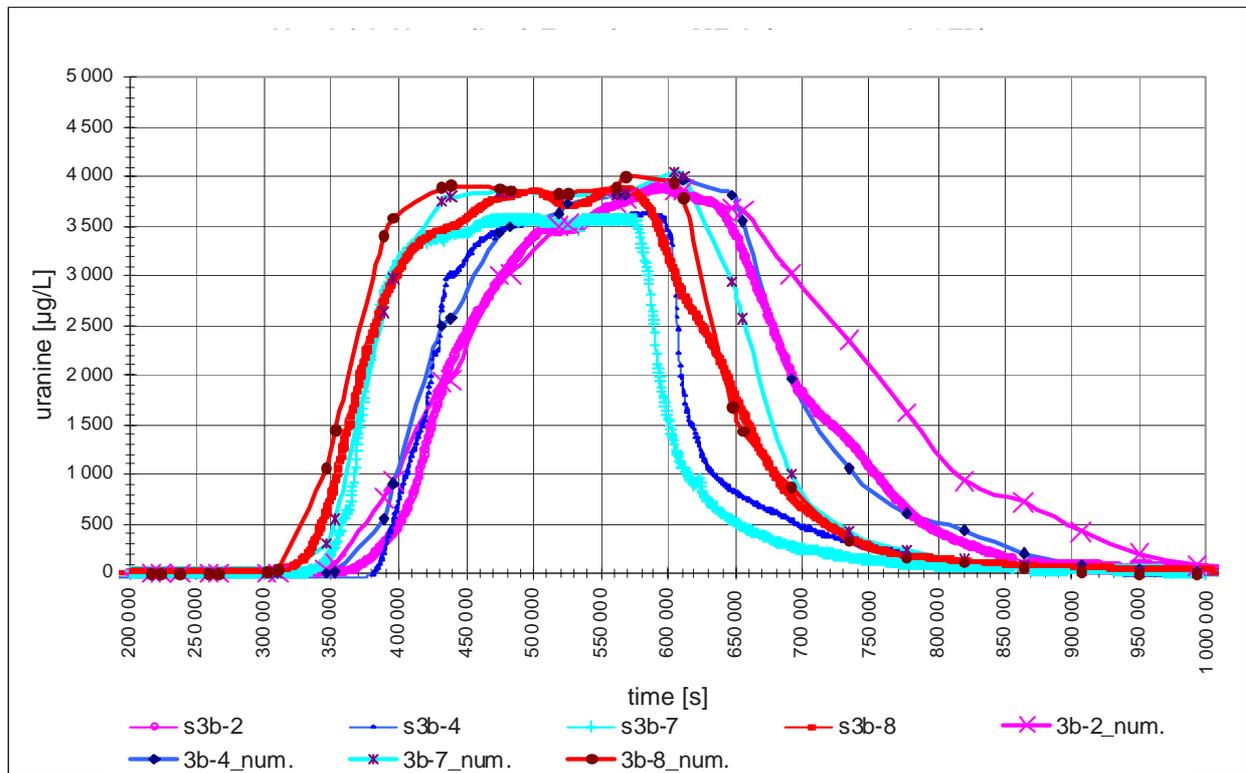


Figure 4: Tracer breakthrough curves at four sampling ports of CP3b (s3b-2, s3b-4, s3b-7, s3b-8) compared to the numerical simulated values (s3b-2_num, s3b-4_num, s3b-7_num, s3b-8_num)

3 Mixing of air and hydrogen peroxide by GCW

Oxygen from the ambient air was added to the circulating groundwater of the GCW by aeration nozzles in a stirring flask. The oxygen saturation ranged between 90 – 95% after the aeration process (~ 2 min) and separation of surplus air. The same set up was used to mix in a 30 % m/m hydrogen peroxide solution to adjust a final concentration of 50 mg/L in the circulating groundwater of GCW. Stoichiometrically, 50 mg/L of hydrogen peroxide result in a maximum concentration of 23.5 mg/L oxygen.

After several days of aeration, aerobic conditions were observed in the Large Flume (Fig. 5). The propagation of the “oxygen front” was close to the travel rate of the groundwater.

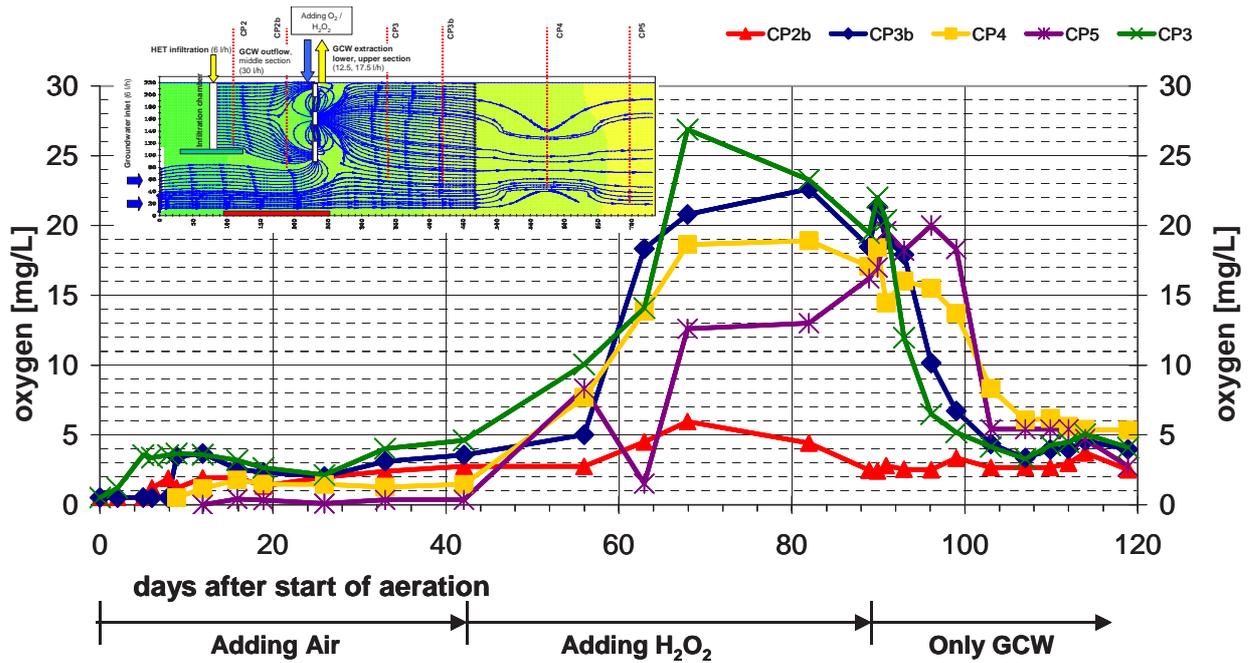


Figure 5: Distribution of oxygen during a) aeration, b) dosing of H₂O₂ and c) stop of infiltration

After 28 days, almost stationary conditions were achieved downgradient of the GCW and oxygen concentrations of 4 mg/L (CP3) and 1.5 mg/L (CP4) were observed. Due to the circulation of groundwater even upgradient of the GCW oxygen concentration up to 2.5 mg/L were observed (ME2b).

Adding hydrogen peroxide (Fig. 5, 42nd day) resulted in a retarded propagation of the “oxygen front” ($R(H_2O_2) \approx 3$). Possible reasons for the retardation are: consumption of oxygen/hydrogen peroxide for aerobic degradation of NSO-HET, oxidation of organic matter (TOC) or reduced minerals (sulfides, Fe(II)) and degassing of the groundwater (air bubbles).

During the infiltration of hydrogen peroxide, the oxygen concentration downgradient of the GCW ranged between 16 – 22 mg/L indicating a stoichiometric release of H₂O and O₂ from H₂O₂. Aerobic conditions were observed upgradient of the GCW (CP2b) showing oxygen concentration > 5 mg/L.

Turning off the electron acceptor infiltration (Fig. 5, 89th day) resulted in an initial fast decrease of oxygen concentration. But it took more than 30 days until the concentrations dropped below 2 mg/L oxygen, indicating anaerobic conditions followed by a significant reduction of microbial activity. It was assumed that the diffusion limited release of oxygen from residual air bubbles in the aquifer (degassed oxygen) caused the comparatively long-lasting period of oxygen concentration of > 2 mg/L.

Monitoring the concentration of hydrogen peroxide downgradient of the GCW a complete decay of hydrogen peroxide was observed within the groundwater circulation. At a distance of 50 cm downgradient of the GCW (2 days of residence time) no hydrogen peroxide was detected whereas oxygen concentrations were > 20 mg/L.. Evidence was found for an instantaneous decay of hydrogen peroxide avoiding regulatory problems in permitting the infiltration of chemicals (hydrogen peroxide).

Batch tests indicated complete aerobic biological degradation of the NSO-HET within 100 days at groundwater temperature. In column tests, the degradation was nearly completed in less than four days of residence time after adaptation of bacteria. The residence time downgradient of the GCW in the Large Flume was approximately 15 days. The travel rate in the Large Flume was reduced in comparison to the field site in order to achieve complete aerobic microbiological degradation of the NSO-HET within the flume.

During batch experiments with original groundwater, dosing hydrogen peroxide to enhance natural attenuation (ENA) processes, it was found that bacteria have to be adapted in order to split off oxygen from hydrogen peroxide. It was expected that starting the aeration process by oxygen will significantly reduce the adaptation time. Therefore the investigations of the ENA processes in the Large Flume started with an initial infiltration of oxygen dissolved in the circulated water of the GCW. After reaching steady-state conditions regarding the oxygen distribution, the infiltration of hydrogen peroxide was started.

Before adding air no significant biological degradation was determined (Fig. 6) but a strong sorption of NSO-HET was observed on the plastic and rubber parts, even on stainless steel filter elements: “mass defect” between “GZB in” and “CP3” (Fig. 6). Sorption was indicated because NSO-HET consisting of three aromatic benzene-rings (dibenzothiophene, acridine) vanished over-proportional compared to 2-ring NSO-HET (6-methyl-quinoline, benzofuran). In general, it was found in batch and column tests that 3-ring NSO-HET are significantly less bio-degradable than 2-ring HET (SAGNER ET AL., 2004).

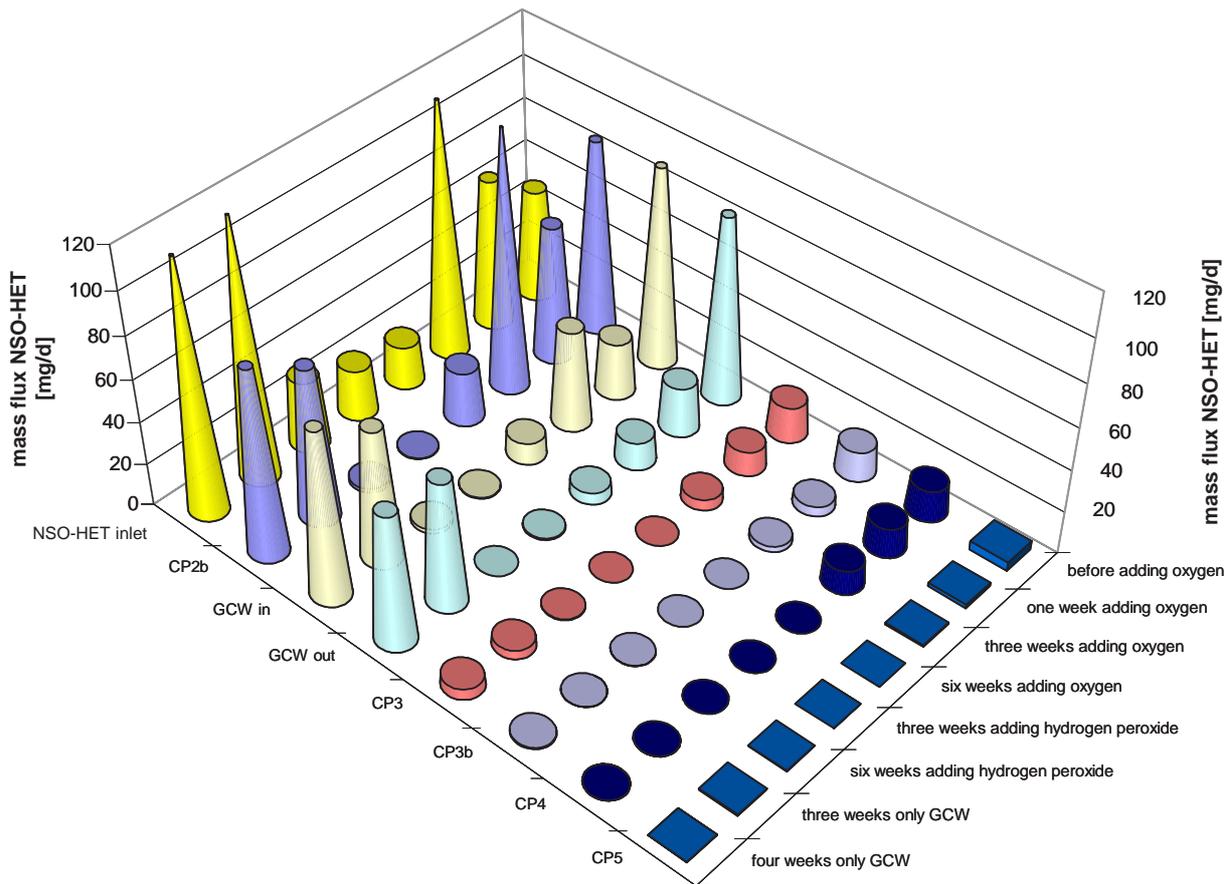


Figure 6: Mass fluxes of NSO-HET in infiltration chamber (NSO-HET in), at different control planes (CP2b, CP3, CP3b, CP4 and CP5) and at inlet and outlet of GCW, a) before aeration, b) during aeration with ambient air, c) during dosage of hydrogen peroxide and d) after stop of dosing

The aerobic biological degradation started immediately after adding air by the GCW. After six weeks of aeration approximately 75% of the contaminant mass flux was degraded (Fig. 6, GCW in vs. NSO-HET inlet). At that moment, the concentration of NSO-HET at CP3 was below the detection limit (1 µg/L). No biological degradation was observed upgradient of the GCW close to CP2b. Even after the reduction of the inlet concentration of NSO-HET (Fig. 6, six weeks air) to meet the contaminant concentration on the field site, no biodegradation was observed under air-adding conditions.

Three weeks after adding hydrogen peroxide, the aerobic degradation increased and even upgradient of the GCW (CP2b) no significant mass flux of NSO-HET was detected and the biological degradation rate was higher than 99%. After another three weeks, the inlet concentrations of NSO-HET were increased and the dosing of hydrogen peroxide was stopped. During the following four weeks, the biological degradation continuously decreased and a mass flux reduction of less than 45 % was measured comparing the inlet mass flux and the outlet of the GCW.

Regarding the kinetic aspects, it was found that the retention time of groundwater in the inner circulation of the GCW, in this case approximately four days, is sufficient for an almost complete aerobic degradation of NSO-HET. Under the given conditions (pH, temperature), assuming first-order kinetics and using the total mass flux of HET instead of concentrations, the degradation coefficient was estimated to be 1.09 1/d for the infiltration of hydrogen peroxide. For the infiltration of air, the coefficient was estimated to be 0.35 1/d.

During the ongoing Large Flume experiment, the boundaries and limitations for a periodical operation of GCW (infiltration of air – infiltration of hydrogen peroxide – no addition of electron acceptors) are currently investigated in order to define the optimal time sequences of each operation mode to maintain sufficient biological degradation and to reduce the costs for chemicals. The results will be transferred to the pilot trial on the test site.

So far the addition of air and in particular the mixing of hydrogen peroxide resulted in a fast and efficient enhancement of the biological degradation of NSO-HET. In the case of hydrogen peroxide addition, an almost complete degradation of the contaminants was observed even upgradient of the well due to the inner water circulation of the GCW.

5 Operation experiences with the GCW

During one year of GCW operation, no significant clogging or precipitation close to the well could be observed. The initial infiltration pressure was 40 mbar. During the aeration of the aquifer with air, the infiltration pressure increased slightly (50 mbar). In the mixing chamber of the GCW, precipitations of iron and manganese were observed. After adding hydrogen peroxide, the precipitations as well as the biofilm dissolved and the infiltration pressure reduced to 30 mbar. After turning off the hydrogen peroxide addition, the pressures increased to the initial value of 40 mbar.

Soil samples taken 10 cm downgradient of the GCW showed no elevated values of iron: 9 mg/kg or manganese: 4.5 mg/kg soil compared to the middle sand directly downgradient of the infiltration chamber. The low iron (<0.2 mg/L) and manganese (< 0.05 mg/L) concentrations in the used water as well as the used quartz sand may have caused the lack of precipitation.

The filter sections of the GCW are made of stainless steel, the packer elements are made of stainless steel and EPDM rubber. So far, no symptoms of material fatigue or corrosion were observed. During a sorption experiment concerning the EPDM rubber, a sorption coefficient of $X_i \approx 0.15 \text{ g / kg}$ was determined for the NSO-HET (2 mg/L), which explains the high sorption effects measured before starting of aeration experiments.

The hydro-geological situation of the field site is dominated by the fluvial system of the Neckar valley: a highly permeable quaternary coarse sandy and gravel aquifer, thickness of 4 m, travel rate of 1-2 m per day.

A couple of numerical simulations using the model approach of the Large Flume showed significant advantages of the GCW in mixing the electron acceptor solutions with the groundwater compared to single- or multi-level well infiltration. Instead of an infiltration of electron acceptors from a point- or line-like source, the electron acceptors are actively mixed over a wider range of the aquifer following the streamlines of the inner circulation of the GCW. The diameter of the inner circulation was numerically simulated for a pumping rate of 15 m³/h to be approximately 10 meters, the same as the extension of the groundwater capturing zone.

The pilot application is planned for the 2nd phase of the project. The funding will be granted within the funding priority KORA and is expected by the summer of 2005. Currently, the planning and the design of the pilot field trial is complete; however, permission for the infiltration of anthropogenic substances (air) and chemicals (hydrogen peroxide) in the quaternary aquifer of the test site in close vicinity to exploited mineral springs still has not been given. Preliminary talks between the scientific board (University of Tübingen (ZAG), TZW and VEGAS), the local consultants, the site owners and the regulators concluded that it is necessary to:

- i) define the risk of formation of metabolites during degradation:
no proofs of metabolites until now,
- ii) determine the effects on the hydrochemistry of the site:
based on simulations using PHREEQC for windows (version 2.8.0.3, D.L.. Parkhurst and C.A.J. Appelo, <http://home.hccnet.nl/vincentpost/phreeqc.html>) the impact can be quantitatively calculated: oxidation of minerals (Fe, Mn), heavy metals and sulfides, shift of CO₃²⁻ / CO₂ equilibrium resulting in resolving Ca and Mg independent of air or hydrogen peroxide infiltration,
- iii) define impact zones for the disintegration / disproportion of hydrogen peroxide:
based on the results of the Large Flume experiments, the disproportion starts immediately after hydrogen peroxide gets in contact with soil / sand and is finalized after two days of residence time (inner circulation of GCW), maximum 4 m downgradient of the GCW in the field case,
- iv) define alarm and surveillance plans as well as define remedies against worse-case scenarios:
weekly measurement of downgradient hydrogen peroxide concentrations, in case of danger: hydraulic containment (GCW solely as extraction well).

Since all requirements for the permission are given, it is expected that permission to infiltrate anthropogenic substances will be obtained within the next few weeks. The activities during the pilot trial are:

- installation of six additional monitoring wells (6") for mass balance, 16 wells are already present at three control planes,
- installation of 15 direct push observation wells in 1 ½" for a detailed NSO-HET concentration monitoring
- installation of a GCW packer system in a present 6" well (B 86, Fig. 7), operation of GCW by extraction of groundwater from the upper filter screen, mixing of air or H₂O₂ and re-infiltration by the lower filter screen,
- multi-tracer tests (uranine, naphthionate) to determine the range of influence of GCW and the hydraulic properties of the aquifer,
- Integral Pumping Tests (CP2-3 and CP2, Fig. 7) for mass balancing before and after pilot application (PTAK T. ET AL., 2003),
- two months of aeration by water jet pump and GCW, eight months of periodical adding of hydrogen peroxide (maximal 25 kg/d of 30% m/m H₂O₂ solution, high technical quality), two months of final periodical aeration.

The numerical simulation of the test site shows a comparatively narrow zone of influence downgradient of the GCW (Fig 7). The dispersion of the oxygen is not yet incorporated. Since the diffusion and

dispersion coefficient of gaseous oxygen are orders of magnitude larger than hydrodynamic dispersion, it is expected that a broader range of influence can be achieved downgradient of the GCW.

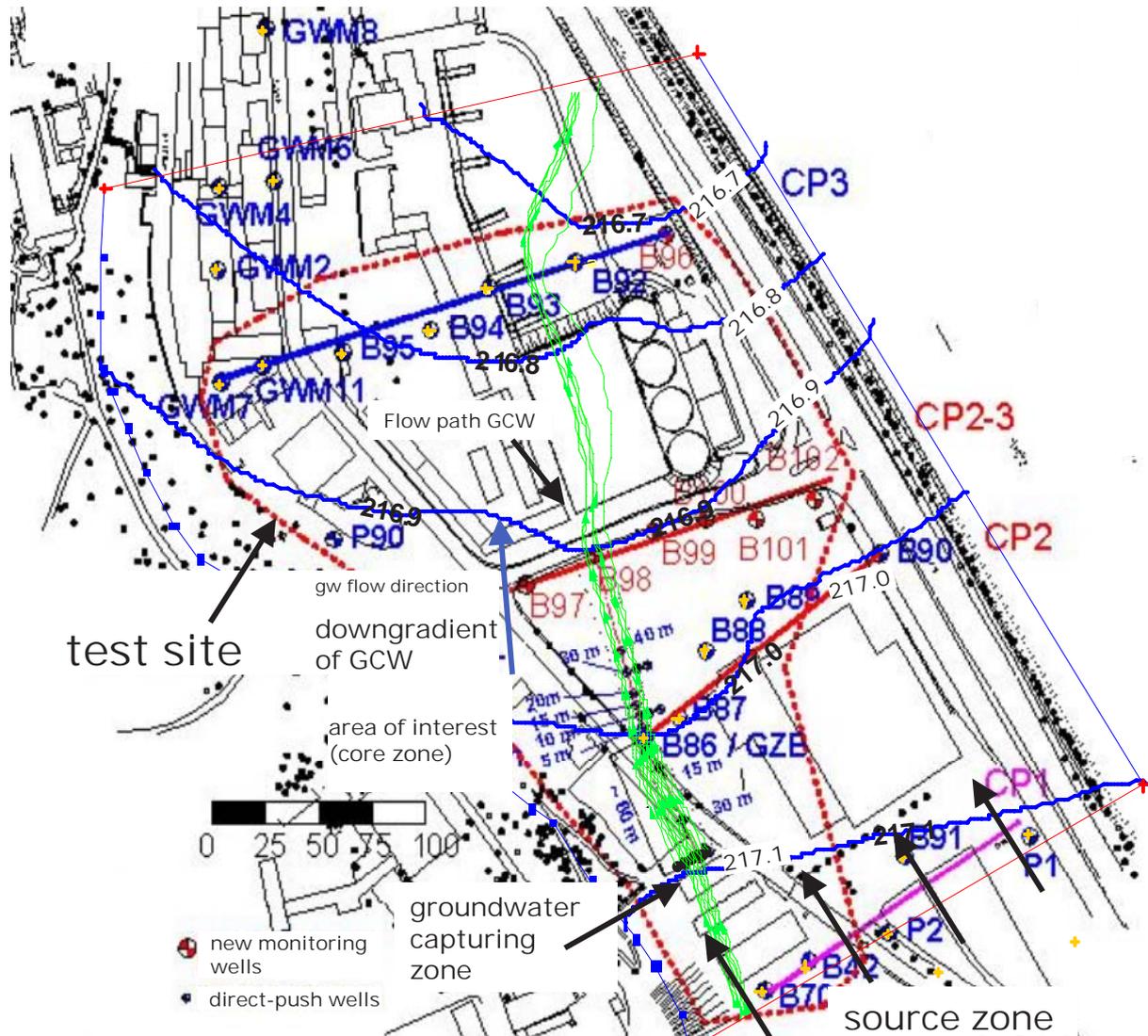


Figure 7: Map of planned field trial, wells location, streamlines of CGW (green), isohypses (blue lines)

7 Summary and conclusions

Heterocyclic hydrocarbons (NSO-HET) are persistent compounds of tar oil residues from former gasworks. In addition, some were found to be highly toxic and carcinogenic. On the basis of the plume leaving the site of a former gasworks it was found that most of the contaminants (PAH, BTEX) are degraded within the plume for about 800 m length except for NSO-HET and benzene. NSO-HET and benzene are biodegradable under aerobic conditions.

A technology for providing bacteria with oxygen was developed and tested in a large physical model: the artificial aquifer in the Large Flume of VEGAS. A Groundwater Circulation Well (GCW) was installed to mix either oxygen from the ambient air or hydrogen peroxide homogeneously in the groundwater contaminated with the relevant NSO-HET from the field site under defined conditions. During the investigations, an almost complete degradation (~ 99%) was determined for both types of electron acceptors. Hydrogen peroxide (50 mg/L) is disproportionated within less than two days into water and oxygen, causing an oxygen concentration of > 20 mg/L. The aerobic degradation of the contaminants is significantly increased, even upgradient of the GCW within the inner circulation of the well system.

Oxygen infiltration was found to be less efficient, limited by the lower concentrations of oxygen (< 5 mg/L) but causes lower costs for the operation than hydrogen peroxide. To reduce costs for the long-term operation of this ENA measure, it is advised to initially create aerobic conditions using hydrogen peroxide release and then switch to aeration in order to maintain the biological degradation. The results of the Large Flume experiment indicate a periodical infiltration of air / hydrogen peroxide. This operation mode has to be optimized first in the Large Flume and later at the field site.

A numerical model using commercially available numerical codes was developed to describe the complex hydraulic system of a GCW in order to transfer the results from the Large Flume to the field site and to design the operation of the GCW to achieve a maximum radius of influence for a minimal pumping rate. Following the simulations a discharge of 15 m³/h will cause a width of upgradient capture of approximately 10 meters.

The test site is located in the Neckar valley. So far, the design and planning of the field trial have been completed. The funding of the field trial is expected by summer 2005. It is planned to install several monitoring and observation wells for mass balancing and hydraulic tests (tracer and pumping tests), one GCW and a treatment unit to mix the electron acceptors. The duration of the field trial is one year. It is planned to start with an initial aeration of the aquifer (two months), then release of hydrogen peroxide (50 mg/L, eight months) and finally a two-month periodical aeration.

Acknowledgement

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