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ASSESSMENT OF INTRINSIC BIODEGRADATION POTENTIALS IN AN AQUIFER CONTAMINATED WITH CHLORINATED ETHENES IN THE VICINITY OF NOWA DEBA

OCENA MOŻLIWOŚCI SAMOISTNEJ BIODEGRADACJI W WODACH PODZIEMNYCH ZANIECZYSZCZONYCH CHLOROWANYMI ETENAMI W REJONIE NOWEJ DĘBY

Abstract: *Natural Attenuation* (NA) in the case of groundwater contaminated with organic compounds relies mainly on intrinsic biodegradation processes. The aim of reliance on natural processes is to achieve site-specific cleanup objectives within reasonable time frames and costs. Such approach may be considered as a risk reduction/remedial option for groundwater contaminated with trichloroethene (TCE) and tetrachloroethene (PCE). This case study presents implementation of the US EPA's guideline "Technical protocol for evaluating natural attenuation of chlorinated solvents in groundwater" to asses intrinsic biodegradation potentials in TCE and PCE contaminated groundwater in the vicinity of Nowa Deba waterworks (south-east Poland). Literature and field data collected from wells and piezometers were used to develop a conceptual model of contaminants' fate and transport from a source to a receptor. The intrinsic biodegradation was investigated basing on available analytical parameters (*eg* concentrations of oxygen, nitrates, chlorides, and pH, TOC and temperature) that are considered as indicators of TCE and PCE transformation. Preliminary screening was done by giving certain points for these parameters, and interpreted in order to asses intrinsic biodegradation potentials. The results indicate inadequate evidence for intrinsic biodegradation (reductive dehalogention) of TCE and PCE, thus a limited potential for NA as a remedial/risk reduction option in the studied case, unless some measures for enhancement of TCE and PCE intrinsic biodegradation are applied.

Keywords: intrinsic biodegradation, TCE, PCE, risk reduction, remediation

Introduction

The term *Natural Attenuation* (NA) refers to the reliance on natural processes to achieve site-specific cleanup objectives within a reasonable time frame and costs [1]. The NA includes a variety of destructive and non-destructive physical, chemical, and biological processes like: dispersion, dilution, adsorption, volatilization, chemical or biological stabilization, biodegradation (biotransformation) or destruction of contaminants [2]. In the case of groundwater contaminated with organic compounds NA relies mainly on intrinsic

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biodegradation processes. Such approach may be considered as a risk reduction/remedial option, or part of an integrated strategy for clean-up of groundwater contaminated with *trichloroethene* (TCE) and *tetrachloroethene* (PCE) in the vicinity of Nowa Deba waterworks (south-east Poland).

TCE and PCE are halogenated alkenes used commonly from the 20's of the last century to the present as industrial solvents. Contamination of groundwater with organic compounds like TCE and PCE have been found worldwide and recently also in several locations in Poland, for example in: Bialogon, Tarnowskie Gory, Nowa Deba (one of the largest TCE and PCE concentration in Poland - recognized by the Inspectorate of Environmental Protection as an "ecological bomb"), Gryfino, Goldap, Srebrna Gora, Budzowie, Lomianki, Poniatowa [3]. The use of drinking water contaminated with TCE and/or PCE can cause nauseas, headaches as well as liver, kidneys and heart problems. *The International Agency for Research on Cancer* (IARC) has passed TCE to the category of 'probably carcinogenic to humans' (Group 2a) and PCE to the 3rd category in terms of carcinogenicity [4]. The limits for potable water are set in Poland at the level of 10 µg/dm³ for the Σ of TCE and PCE [5]. Because of the scale of the TCE and PCE contamination looking for remedial measures is necessary.

Generally, TCE and PCE may be present both, as: DNAPLs (*denser* than water *non-aqueous phase liquids*) and dissolved in groundwater [1]. They are denser than water (1.465 g/dm³ for TCE and 1.623 g/dm³ for PCE) with solubility of 1100 mg/dm³ (TCE) and about 150 mg/dm³ (PCE) [6]. Biodegradation is usually the most important destructive process leading to reduction of the contaminant loads in groundwater, therefore it is an important factor to consider [7]. The evaluation of in situ biodegradation is essential for the implementation of NA concepts in groundwater management strategies [1]. The biodegradation of TCE and PCE may occur firstly by reductive dehalogenation in anaerobic conditions or secondly by cometabolism in aerobic conditions. Reductive dehalogenation of TCE and PCE is associated with the accumulation of *daughter products* (DCE and *VC*) and vinyl chloride increase in the chloride concentration [7].

To estimate the intrinsic TCE and PCE biodegradation rates different lines of evidence can be used [8]. Microcosm studies can physically demonstrate that NA is occurring but they are time consuming and expensive [7]. The in situ degradation of chlorinated ethenes can be also assessed using a taxon specific detection of specific dehalogenation groups of bacteria. In recent years, stable isotope fractionation approaches have been developed for the assessment of in situ biodegradation processes. Stable carbon isotope fractionation of chlorinated ethenes has been shown to occur in situ [9]. All above-mentioned methods are effective, however access to properly equipped laboratories and a very good theoretical background are crucial to carry out such studies. An effective alternative is to base the estimation on data about groundwater and soil chemistry (biogeochemistry and hydrogeochemistry), as well as on hydrogeological conditions.

To evaluate whether NA by itself, or in conjunction with other remedial technologies is sufficient to achieve site-specific remedial objectives as concerns TCE and PCE contaminated groundwater in the vicinity of Nowa Deba, the USEPA's guideline "Technical protocol for evaluating natural attenuation of chlorinated solvents in ground water" [7] called further the Protocol was implemented. Examples of implementation of this protocol can be found elsewhere [8, 10, 11].

Methodology

The Protocol was designed to evaluate the fate of chlorinated aliphatic hydrocarbons in groundwater [7]. It is aimed at improving the characterization process for sites at which a remediation involving *monitored natural attenuation* (MNA) is being considered. It contains methods and recommended strategies for completing the remedial investigation process. Emphasis is placed on developing a more complete understanding of the site through the conceptual site model, early pathways analysis and evaluating potential remediation processes. Understanding the contaminant transport in the subsurface is essential for a technically justified evaluation of the MNA-based remedial option.



Fig. 1. Screening process flow chart (after [7, 10])

According to the Protocol, firstly available data about site (site history, groundwater and soil chemistry, hydrogeological conditions) and contamination must be collected. Basing on that the conceptual model can be developed. Next, the assessment process, which involves a six-step screening method (Fig. 1) can be performed [10].

Data collection

Site data were collected from archive documentation and field studies. Groundwater was collected mainly in October and November 2011 and supplemented in July 2011, according to PN-ISO 5667-11:2004. Field parameters like: *dissolved oxygen* (DO), *oxidation-reduction potential* (ORP), conductance [12], pH, temperature, were measured in situ during groundwater sampling. Groundwater samples were also analyzed in the field for chlorides and alkalinity [13].

Samples for organic constituents were collected in head-space free glass vials, kept cool (4°C) until delivery to the laboratory. Analyses were conducted in the accreditated accordance with [14] and [15]. Samples for inorganic constituent were collected in PE bottles, transported in the same manner and analyzed in the accreditated laboratory of the Department of Hydrogeology and Engineering Geology at AGH University of Science and Technology in Krakow. Analyses were performed in accordance with [16-18] using gas chromatography and mass spectrometry (ICP-MS - Elan 6100 and ICP-OES - OPTIMA 7300dv from Perkin Elmer). The concentration of iron(II) was calculated using PHREEQC (Version 2.15.0), while contour maps were generated using Surfer[®] (Golden Software, Version 9).

Intrinsic biodegradation assessment

Assessing the intrinsic biodegradation potentials is based on assigning a specified number of "points" depending on the concentration or value of the geochemical indicators of TCE and PCE transformation (DO, nitrates, chlorides, pH, TOC, temperature) observed in the wells. The "points" can be awarded only if the concentration and value of a geochemical indicator (see Table 2) is within the range specified in the screening criteria, and if the indicator is not a constituent of the original contaminant source. The "points" are added and interpreted to determine whether biodegradation is occurring at the selected location [10]. Table 1 presents the interpretation of given "points".

Table 1

Score	Interpretation
0 to 5	Inadequate evidence for anaerobic biodegradation (RD) of chlorinated organics
6 to 14	Limited evidence for anaerobic biodegradation (RD) of chlorinated organics
15 to 20	Adequate evidence for anaerobic biodegradation (RD) of chlorinated organics
> 20	Strong evidence for anaerobic biodegradation (RD) of chlorinated organics

The interpretation of "points" for MNA (via reductive dehalogenation RD) assessment process [7]

Three points were selected to characterize the Nowa Deba site: a piezometer M-5 located about 1100 m from source area, and two wells: S-2tr (about 900 m from source area) and S-6b (about 2400 from source area) (see Fig. 2). Preliminary screening was done by giving certain points for each parameter, and interpreted in order to assess intrinsic biodegradation potentials according to the scoring scheme provided by Wiedemeier et al [7].

Characteristics of biogeochemistry involves description of range of geochemical indicators, briefly discussed below. The DO level informs about aerobic/anaerobic conditions in the aquifer. Nitrates and sulfates concentrations below background values in areas with high organic carbon concentrations are indicative of denitrification and sulphate

reduction, respectively [7]. Iron(II) is being produced at a site, where reductive dechlorination of alkenes is ongoing. The optimal pH range for reductive biodegradation is 5-9. The ORP may indicate if reductive processes are likely to occur. Total organic carbon (TOC) informs about the carbon content in the aquifer material that is an energy source and drives dechlorination. Temperature ($T > 20^{\circ}C$) is also important, because at higher temperature biochemical processes are accelerated. Dichloroethene (DCE) or vinyl chlorides (VC) are metabolites of TCE and PCE biotransformation, and their presence (if not released at a site) indicates that reductive dehalogenation may take place.

Results and discussion

Site and contamination characteristics

In Nowa Deba (S-E Poland) the metal works "DEZAMET" established in 1939, firstly as an ammunition factory, have been operating until now in a restructured form. TCE and PCE were used there for degreasing the metal parts.

Hydrogeological conditions

Groundwater is connected with the Quaternary river deposits, which consist mostly of sands, gravels and silt-clay intermediate layers. The most permeable deposits occur in the bottom part of the aquifer. The Quaternary deposits attain thickness of over 30 m and are underlain by impermeable Miocene clays. The aquifer is unconfined and depths to groundwater vary from 1.0 to 9.0 m. The recharge proceeds through direct infiltration. The drainage occurs naturally via streams. Current flow conditions are modified due to groundwater extraction by municipal waterworks). This aquifer is the main source of potable water in the Nowa Deba region, and the groundwater flow is predominately from south-east towards the waterworks.

Contamination

The contaminants' distribution maps (Fig. 2) show that the source of TCE and PCE is located within the area of the former metal works "DEZAMET". The contaminants have probably been releasing gradually for decades into soil and groundwater because of *eg* improper storage and usage with no or poor environmental concern. TCE has already reached wells of the municipal waterworks while PCE is less widespread.

Unfortunately, the release area (or areas) is not accurately delineated, however the contaminants' distribution maps show that the source is located within the area of the former metal works "DEZAMET". The period when contaminants of concern were released and, if the release is already stopped, are also open questions to be answered.

High TCE and PCE concentrations of up to 6130 and $694 \mu g/dm^3$, respectively, observed in some wells of the waterworks [19] exceed the limits for potable water [5]. Moreover, they are responsible for the "poor" chemical status of groundwater (class V) in the Nowa Deba region according to [20] (Fig. 3). Although groundwater contamination with TCE and PCE is considered to be the most important problem in the studied area, the poor chemical status is caused also by the exceeded permissible values of pH, as well as concentrations of iron, nickel and aluminum [19].



Fig. 2. Distribution of: TCE and PCE in groundwater [µg/dm³] in the vicinity of Nowa Deba

A conceptual model

A conceptual model of TCE and PCE fate and transport in groundwater in the vicinity of Nowa Deba (Fig. 4) was developed based on the "source - pathway - receptor" sequence [2]. The source is within the area of the former metal works "DEZAMET", while the municipal waterworks in Nowa Deba, located down gradient about 2000 m from the alleged source, supplying about 20 000 inhabitants with potable water, is the receptor affected by the contamination. The wells in which contaminations were detected have been excluded from operation. Currently, as a temporary measure TCE and PCE is partially discharged into atmosphere in three splattering points consisting of perforated pipes, which decreases slightly the risk for the receptor but causes emissions of vapors into the atmosphere.

TCE and PCE migrate in the subsurface vertically depending upon gravity and capillary forces through the unsaturated zone. If the released volume is significant, they may reach and penetrate the groundwater table as a result of high density, dissolve and form a solute plume. Within the aquifer, a certain quantity of solvents may be entrapped as blobs or forms pools over low permeability geological structures. Infiltration of DNAPLs is not uniform, and is influenced by both the heterogeneity of the porous medium and the difference of viscosity between water and solvents, what generates fingering. TCE and PCE may migrate via preferential pathways. Dissolution of DNAPLs is not homogeneous in space and seems to be rate limited. It depends on the: groundwater flow rate, shape of the source zone and interfacial area between organic and aqueous phases [7, 21]. Along the transport pathway a variety of physical, chemical or biological processes influence contaminants' migration. Those processes include: sorption, dispersion, dilution, volatilization and biodegradation.



Fig. 3. Map of the study area with point evaluation of the chemical status of Quaternary groundwater (after [19], changed). 1 - main groundwater flow direction; 2 - former metalworks "DEZAMET" site; 3 - surface water courses; 4 - trenches with insolated beds; 5 - wells and piezometers; 6-9 - color code of groundwater quality classes (6 - class II, 7 - class III, 8 - class IV, 9 - class V)



Fig. 4. Conceptual model of contaminants' fate and transport

Characteristics of biogeochemistry

Dissolved oxygen

Basing on DO levels it can be suspected that there are large anaerobic areas within the studied site. High DO contents are observed only close to the source area and in the vicinity of waterworks wells (Fig. 5A). Generally, the DO content is below 0.5 mg/dm³.

Alkalinity

The background level for alkalinity expressed as $CaCO_3$ is 12-62 mg/dm³ (Fig. 5B). There is a positive correlation between zones of microbial activity and increased alkalinity. Increaseds in alkalinity result from the dissolution of rock driven by the production of carbon dioxide as a result of the metabolism of aerobic microorganisms [7]. In the described case higher values of alkalinity are observed in the central part of the plume.

Chloride

The background level for chloride is $12-52 \text{ mg/dm}^3$. Higher chloride concentrations are observed in the contaminated area (Fig. 6A). In the southern (against groundwater flow direction) and northern (where contaminations have not arrive yet) part of the contaminants plume, chloride concentration is below 15 mg/dm^3 , while in the central part it reaches the level of 50 mg/dm³ and more.



Fig. 5. Distribution of: (A) DO and (B) alkalinity [mg/dm³] in groundwater in the vicinity of Nowa Deba



Fig. 6. Distribution of: (A) chloride and (B) sulfate [mg/dm³] in groundwater in the vicinity of Nowa Deba

Nitrate, iron and sulfate

Concentrations of additional terminal electron acceptors (nitrate, sulfate) are higher than the highest values allowed in the Protocol (Fig. 6B). Dissolved iron(II) levels are diversified across the area of concern, what suggests that iron reduction is not prevalent over some part of site with lower concentrations of iron(II).



Fig. 7. Distribution of: (A) ORP [mV] and (B) pH [-] in groundwater in the vicinity of Nowa Deba



Fig. 8. Distribution of: (A) TOC [mg/dm³] and (B) temperature [°C] in groundwater in the vicinity of Nowa Deba

Oxidation Reduction Potential (ORP), pH, TOC, temperature

The ORP distribution (Fig. 7A) indicates that reductive dehalogenation pathway is not taking place. In the Protocol ORP is expressed as the potential against the Ag/AgCl

electrode. pH is in optimal range for reductive dehalogenation pathway in some distance from the contamination source (Fig. 7B). Around the source area measured pH values are in the range of 4-6. The TOC content, which can be natural or anthropogenic (Fig. 8A) is rather low. Knowledge of the TOC content of the aquifer matrix is important for sorption and solute-retardation calculations [7]. Basing on TOC values, it can be suspected that sorption does not play a significant role in retardation of contaminants' transport. The temperature criterion (> 20°C) cannot be fulfilled normally under the climate conditions in Poland for Quaternary groundwater that is also the case at the described site (Fig. 8B).

Daughter products

Any daughter products of TCE and PCE like dichloroethene (DCE) or vinyl chloride (VC) biotransformation were detected. TCE can be a product of PCE biotransformation, however it was one of the material released.

Intrinsic biodegradation assessment

The field data collected from three wells located in different parts of the plume were used to complete a biodegradation scoring analysis recommended by the Protocol. The results of the intrinsic biodegradation assessment are summarized in Table 2.

Table 2

	Concontration/Value		M-5		S_2tr		S-6h	
Analysis	in the Most Contaminated Zone	Value	Concent./ Value	Score	Concent./ Value	Score	Concent./ Value	Score
Oxygen	$< 0.5 \text{ mg/dm}^3$	3	0.42	3	4.60	0	0.23	3
Nitrate	$> 5 \text{ mg/dm}^3$	-3	8 58	0	4 75	0	14.8	0
Iron(II)	$> 1 \text{ mg/dm}^3$	3	0.03	0	0.48	0	2.11	3
Sulfate	$< 20 \text{ mg/dm}^3$	2	76.5	0	113.0	0	88.8	0
Oxidation Reduction	< 50 mV	1	111		124	0	-5	1
Potential (ORP) against Ag/AgCl electrode	< -100 mV	2		0				
лЦ	5 < pH < 9	0	6.5	0	6.2	0	6.4	0
рн	5 > pH > 9	-2						
TOC	$> 20 \text{ mg/dm}^3$	2	3.7	0	2	0	1.1	0
Temperature	> 20°C	1	11.7	0	11.9	0	9.8	0
Alkalinity	> 2x background	1	147.3	1	51.2	1	0.0	0
Chloride	> 2x background	2	53.6	2	31.9	0	18.5	0
BTEX	$> 0.1 \text{ mg/dm}^3$	2	NA	0	ND	0	NA	0
PCE [$\mu g/dm^3$]		0	147	0	243	0	ND	0
TCE [µg/ dm ³]		0 2 ^{a/}	721	0	631	0	149	0
VC [µg/ dm ³]		0 2 ^{a/}	NA	0	ND	0	NA	0
Total points awarded				6		1		7

Analytical parameters and weighting for preliminary screening for anaerobic biodegradation processes^{a/} (after [7], changed)

^{a/} Points awarded only if it can be shown that the compound is a daughter product (ie, not a constituent of the source NAPL). NA - not analyzed, ND - not detected

In selected locations the score is between 0 and 7 points, what indicates: "Inadequate/limited evidence for anaerobic biodegradation of chlorinated organics" (see Table 1). Points were assigned to two locations due to low concentration of DO. They were also assigned for high alkalinity and iron(II). It should be noted, however, that not all parameters listed in the Protocol (*eg* hydrogen, carbon dioxide, ethane, sulfide) could be rated. But it seems that they should not interfere significantly the assessment result.

Conclusions

Different lines of evidence can be used to estimate the intrinsic TCE and PCE biodegradation rate, including: microcosm studies, taxon specific detection of dehalogenation groups of bacteria, stable isotope fractionation approaches. These methods are effective, however time-consuming and expensive. An effective alternative is to base the estimation on data about groundwater and soil chemistry (biogeochemistry and hydrogeochemistry), as well as on hydrogeological conditions. Such estimation does not allow for accurate results but it can be used as first approximation. In some cases such accuracy is good enough, and there is no need to perform any additional, sophisticated tests to determine biodegradation rates.

The Protocol was implemented in the area of Nowa Deba to answer the question: Is biodegradation occurring? Intrinsic biodegradation of TCE and PCE via reductive dehalogenation was shown not to be enough efficient for risk reduction in this case. Therefore, the remedial option to achieve site-specific objectives cannot be based exclusively on MNA. However, additional investigations must be performed, *eg*: batch and column tests, to find sorption parameters, retardation coefficients that can be used in numerical modeling to establish evidently contaminants mass transport in the aquifer. Natural processes have to be probably enhanced using *in situ remediation* (ISR) methods, like *eg permeable reactive barriers* (PRB) or *in situ chemical oxidation* (ISCO).

Detailed description of site biogeochemistry and hydrogeochemistry along with the developed conceptual contaminants' fate and transport model were proved to be valuable tools in assisting selection of effective remediation measures for the studied case.

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OCENA MOŻLIWOŚCI SAMOISTNEJ BIODEGRADACJI W WODACH PODZIEMNYCH ZANIECZYSZCZONYCH CHLOROWANYMI ETENAMI W REJONIE NOWEJ DĘBY

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Abstrakt: Samooczyszczenie (ang. *natural attenuation* - NA) w przypadku wód podziemnych zanieczyszczonych substancjami organicznymi, polega głównie na samoistnej biodegradacji zanieczyszczeń. Oparcie remediacji na naturalnych procesach zakłada osiągnięcie wymaganych efektów oczyszczania specyficznych dla danego terenu przy zachowaniu rozsądnych ram czasowych i kosztów. Takie podejście może być zastosowane jako jedna z opcji likwidacji zagrożenia/remediacji wody podziemnej zanieczyszczonej trichloroetenem (TCE) i tetrachloroetenem (PCE) w rejonie ujęcia Nowa Dęba (południowo-wschodnia Polska). Do oceny możliwości samoistnej biodegradacji TCE i PCE w rejonie Nowej Dęby wykorzystano procedurę opisaną w dokumencie US EPA

"Technical protocol for evaluating natural attenuation of chlorinated solvents in ground water". Dane archiwalne oraz wyniki badań terenowych zostały wykorzystane do opracowania modelu koncepcyjnego transportu zanieczyszczeń od ogniska do receptora. Samoistną biodegradację oceniono na podstawie dostępnych parametrów analitycznych (np. stężenia tlenu, azotanów, chlorków oraz pH, TOC i temperatury wody), które uważane są jako wskaźniki rozkładu TCE i PCE. Ocena polegała na przypisaniu odpowiedniej liczby punktów i wag dla poszczególnych parametrów oraz interpretacji uzyskanych wyników w celu sprawdzenia potencjalnych możliwości samoistnę biodegradację redukcyjną) TCE i PCE w wodach podziemnych w rejonie ujęcia Nowa Dęba. Wynika z tego, że w opisywanym przypadku oparcie remediacji wód podziemnych (i likwidacji zagrożenia dla ujęcia wody) na NA jest możliwe jedynie pod warunkiem zastosowania metod wspomagających biodegradację TCE i PCE.

Słowa kluczowe: samoistna biodegradacja, TCE, PCE, likwidacja zagrożenia, remediacja