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Applying the Triad method in a risk assessment of a former surface treatment and metal industry site

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A B S T R A C T

With a greater focus on soil protection in the EU, the need for ecological risk assessment tools for cost-effective characterization of site contamination is increasing. One of the challenges in assessing the risk of soil contaminants is to accurately account for changes in mobility of contaminants over time, as a result of ageing. Improved tools for measuring the bioavailable and mobile fraction of contaminants is therefore highly desirable. In this study the Triad method was used to perform a risk characterization of a former surface treatment and metal industry in Eskilstuna, Sweden. The risk assessment confirmed the environmental risk of the most heavily contaminated sample and showed that the toxic effect was most likely caused by high metal concentrations. The assessment of the two soil samples with low to moderate metal contamination levels was more complex, as there was a higher deviation between the results from the three lines of evidence; chemistry, (eco)toxicology and ecology. For the slightly less contaminated sample of the two, a weighting of the results from the ecotoxicological LoE would be recommended in order to accurately determine the risk of the metal contamination at the sampling site as the toxic effect detected in the Microtox® test and Ostracodtoxkit™ test was more likely to be due to oil contamination. The soil sample with higher total metal concentrations requires further ecotoxicological testing, as the integrated risk value indicated an environmental risk from metal contamination. The applied methodology, the Triad method, is considered appropriate for conducting improved environmental risk assessments in order to achieve sustainable remediation processes.

1. Introduction

A greater focus on soil protection in the EU [1] in combination with the ever-growing pressures of redevelopment of contaminated and former brownfield sites, the need for ecological risk assessment tools for cost-effective characterization of site contamination is increasing.

One of the greatest challenges in assessing the actual risk of soil contaminants is to accurately account for the reduced mobility of contaminants over time, as a result of ageing [2,3]. Improved tools for measuring the bioavailable and mobile fraction of the contaminants, as opposed to total concentrations, is therefore highly desirable. The bioavailable fraction of a contaminant can be defined as the "toxicologically available fraction" [4] and it is therefore of great importance that it is taken into account in ecological risk assessments [5]. Factors that affect the bioavailability of soil contaminants include contaminant chemical properties, such as sorption ability, persistence, degradability and water/lipid solubility; soil characteristics, e.g. amount and type of organic carbon, particle size and type; and the ecology of the soil dwelling organisms [6]. The bioavailability of the soil contaminants being unknown, great uncertainties has to be accounted for when comparing total soil contaminant concentrations with benchmark values. This could in turn lead to an overestimation of the soil contaminant exposure levels [7], with incorrect decisions of clean-up procedures being made. This is neither environmentally nor socio-economically favorable.

Several countries and regions have developed methodologies for performing ecological risk assessments. USA, Canada and the Netherlands are leading the development of ecological risk assessments. The US EPA methodology for ecological risk assessment, the Superfund Program [8], aims to quanitify both potentially adverse effects to humans and the ecological risks at contaminated sites.

In Sweden, the Environmental Protection Agency has revised the model for calculating guideline values and published new general guideline values for contaminated soil together with a calculation program for site-specific guideline values [9]. This is a move away from a generic approach using benchmark values, towards a site-specific risk assessment approach. Guidelines for performing site-specific assessments of the soil ecology system have not

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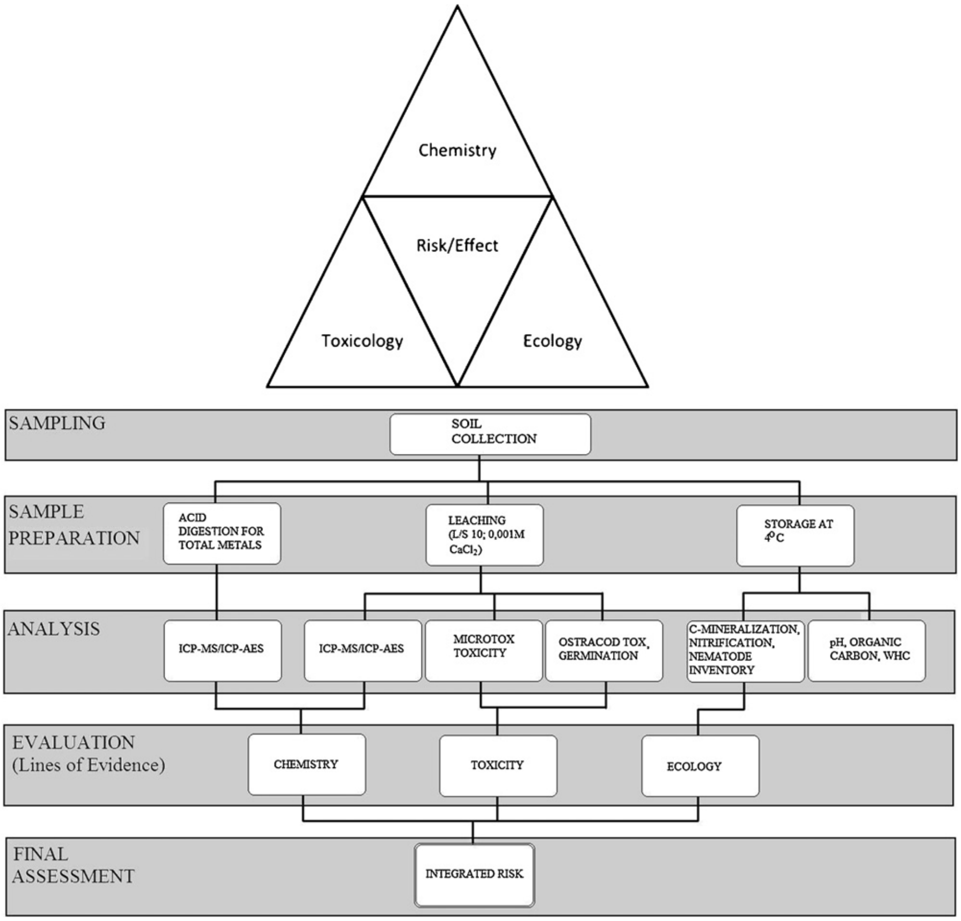

Fig. 1. (a) A schematic presentation of the integration of the three lines of evidence, LoE, chemistry, toxicology and ecology using an weight-of-evidence, WoE, approach in the Triad risk assessment method. Adapted from Jensen and Mesman (2006). (b) An overview of the Triad risk assessment process in the study. The risk assessment is performed in five steps, with simultaneous measurements of chemical, toxicological and ecological parameters of metal contamination. The results within (intra) and between (inter) LoE are then integrated in order to determine the environmental risk at the site.

been developed, which is problematic as this in many cases is the determinant for the calculated guideline values.

The research project Liberation, supported by the European Commission under the Energy, Environment and Sustainable Development Program [10], aimed at developing a decision support system for ecological risk assessment of contaminated sites. As part of the decision support system, a tiered system, including the Triad method [11], was proposed for the later stages of the risk assessment. The Triad approach is based on the Sediment Quality Triad [12], developed in the late 1980s for sediment quality assessment purposes. This method integrates contaminant chemistry and bioavailability analyses with observed ecotoxicological effects in soil and groundwater. It enables a quantification of the ecological risk through a weight of evidence (WoE) approach [13–15], combining the three lines of evidence (LoE); chemistry, toxicology and ecology, see Fig. 1 [6].

The major assumption when using a WoE approach in ecological risk assessment is that a conclusion about potential ecological effects drawn from several independent lines of enquiry will decrease the uncertainty in the risk assessment decision-making [6]. Using the Triad approach, a variety of chemical, (eco)toxicological and ecological analyses or tests of increasing complexity and specificity are employed in a tiered assessment process, wherefrom a final number of risk is determined. This subsequent integration of the results within and between the three LoE may, however, be seen as a comparison of incommensurables. Furthermore, although a quantification of ecological risk is performed it may still be impossible to decide on further action since a high deviation between the different LoE may necessitate additional research.

In the present study, a risk characterization of a former surface treatment and metal industry in Eskilstuna, Sweden was performed. On the site, high concentrations of copper, zinc and nickel have been detected in the soil in an earlier investigation of contaminant levels.

The aim of the present study was to apply the Triad method for a site-specific risk assessment of the above mentioned Swedish industrial site.

For performing the risk assessment of the site according to the Triad method, contaminant bioavailability, soil metal concentrations and soil toxicity were determined and an ecological assessment was carried out on four selected soil samples (one reference soil and three samples with low, medium and high contaminant levels).

2. Materials and methods

The procedure for this Triad risk assessment is shown in Fig. 2. Contaminant bioavailability was measured through leaching and biouptake tests (DGT). Metal concentrations in soil samples (acid digestion) and soil leachates were determined by ICP-MS. Ecotoxicity of the soil samples was measured in luminescent bacterial assays, whole soil direct-contact tests with a meiobenthic ostracod and germination/growth inhibition tests using radish and white

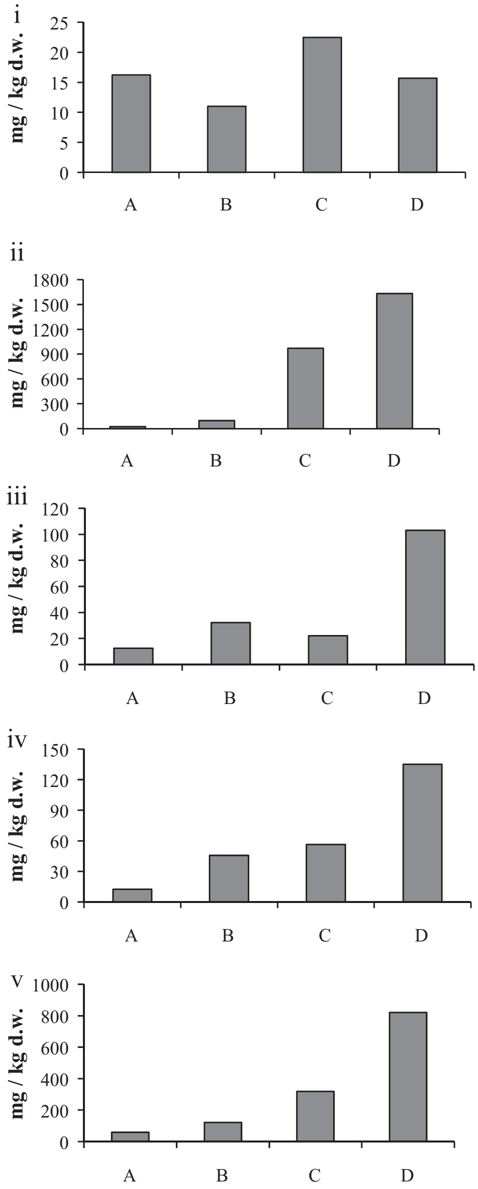

Fig. 2. (i–v) Total metal concentrations in soil (acid digestion) in mg/kg d.w. at the four sampling points, A, B, C and D. The metals analyzed were; Cr (i), Cu (ii), Ni (iii), Pb (iv), and Zn (v).

clover seeds. The assessment of the ecological status of the soil samples was performed by determination of microorganism C-mineralization, nitrification rates. A nematode inventory [16] was performed as part of this study, it was however, withdrawn form the subsequent risk assessment due to test failure. The sampling conditions and the analytical method were not optimal for nematode survival, therefore any nematodes which may have been present in the sample could have died prior to the termination of the test. As the test is dependent of live nematodes, no clear conclusions could be drawn from the test and it was therefore not included in the risk calculations.

2.1. Sampling

Sampling was performed at the site based on contamination history and with the aid of XRF (X-ray fluorescence), four suitable sampling points, with varying metal concentrations, were localized. The sampling points were selected according to contamination levels; point A = reference point, background contamination levels; point B = low metal contamination levels, considered for remediation; point C = moderate metal contamination levels, considered for remediation; and point D = high metal contamination levels, area to be remediated.

Sampling was divided into two different *z*-coordinates; 0–0.1 m and 0.1–0.7 m. Soil samples from the surface were collected with a spade, while sampling at the higher depth was performed with a spade and a crowbar. At all sampling points, randomly selected soil samples from the dug up soil were collected into a pooled sample.

The reference sample was selected from a point where no elevated metal concentrations were detected by XRF. This sample was set as the point with no expected environmental risk, in order to enable a comparison with the samples with elevated levels of metal contamination. If any of the analyses of the contaminated samples showed (marginally) lower risk than the reference point, the risk was raised to zero risk. This was motivated by the fact that all values should lie within the range set; 0–1, where 0 = no risk, and 1 = maximum risk. This also minimised the risk of underestimating risks.

2.2. Sample preparation

Prior to dividing the samples for the different analyses, samples were sieved at the sampling point. Samples for C-mineralization and the diffusive gradient in thin film (DGT) biouptake tests were immediately sent of for analysis at external certified laboratories, while remaining samples were kept at 4 °C until analysis.

2.3. Chemical analysis

All chemical reagents used in the chemical analyses and toxicity tests were of analytical grade. Physicochemical soil parameters such as particle size, pH, WHC (water-holding capacity), dry weight and loss-on-ignition were determined.

The soil samples for the total metals analysis were acid digested in 7 M HNO_3 (Suprapur) at 170 °C for 30 min according to Swedish standard SS 02 83 11 [17]. Subsequently, the samples were decanted and sent off for analysis at the certified laboratory ALS Analytica.

A soil leaching test was conducted according to standard procedures ISO 21268:2 [18].

The diffusive gradient in thin film (DGT) test was performed by ALS analytica, according to a method described by Zhang [19].

2.4. Toxicity bioassays

The acute toxicity of unpreserved leachate samples to *Vibrio fischeri* was assessed using the Microtox® ISO 11348-3 [20] standard procedure.

The acute and chronic ostracod toxicity tests were performed with whole soil samples according to the Ostracodtoxkit™ standard operational procedures [21], using the meiobenthic crustacean *Heterocypris incongruens*.

The germination/growth inhibition test with radish and white clover was performed according a method published by IVL [22].

2.5. Soil ecological status tests

The C-mineralization test was performed by the Department of Ecology at the Swedish University of Agricultural Sciences, according to a method described by Persson et al. [23].

The nitrification test was performed according to standard procedures ISO 13395 [24].

2.6. Calculation of risk according to the Triad method

A number of mathematical scaling operations were performed in this study to enable a quantification of the risk. Moreover, integration operations were undertaken in order to combine the different test results in the Triad risk assessment [6].

The scaling of the chemical analysis results were conducted according to the following:

$$R_1 = \frac{1}{1 + \exp(\log(\text{guide value}) - \log(\text{result}))\beta}$$

$$R_2 = \frac{R_{1\,\text{sample}} - R_{1\,\text{reference}}}{1 - R_{1\,\text{reference}}}$$

$$R_3 = 1 - ((1 - R_2)1 \cdot (1 - R_2)2 \cdot (1 - R_2)3 \cdot \cdots \cdot (1 - R_2)n)$$

The scaling of the ecotoxicological analysis results were conducted according to the following:

$$R_1 = \frac{100 - \text{result}(\%)}{100}$$

$$R_2 = \frac{R_{1\,\text{sample}} - R_{1\,\text{reference}}}{1 - R_{1\,\text{reference}}}$$

The scaling of the soil ecological test results were conducted according to the following steps:

$$R_1 = \frac{\text{result}}{\text{reference value}}$$

$$R_2 = |\log R_1|$$

$$R_3 = -1 \cdot \Sigma(R_2)n$$

$$R_4 = n(\text{number of results})$$

$$R_5 = 1 - 10^{(R_3/R_4)}$$

The integration of the results from tests within an LoE was conducted according to the following:

$$1 - ((1 - R_1) \cdot (1 - R_2) \cdot (1 - R3) \cdot \cdots \cdot (1 - R_n))^{1/n}$$

The calculation of the final, integrated risk between all the LoE were conducted according to the following:

$$1 - ((1 - R_1) \cdot (1 - R_2) \cdot (1 - R3) \cdot \cdots \cdot (1 - R_n))^{1/n}$$

3. Results

The chemical analysis showed similarities between the previously measured XRF levels and the total metal concentrations in acid digested soil samples, as shown in Fig. 2i–v. The pattern of contamination was as expected; the reference sample, A, had the lowest metal concentration, while sample D, sampled from an area planned to be remediated, showed the highest metal concentrations. There has been discussions about the possible remediation of sampling points B and C, as total metal concentrations have been shown to be elevated in earlier analyses. This analysis showed relatively high concentrations of zinc at sampling point B and of both copper and zinc at point C.

Analysis of the soil leachates revealed a correlation of the soil leachate metal concentrations and the total metal soil concentrations. However, the highest leachable fraction for all metals was found in the reference sample, A.

The results from the DGT analysis did not show any clear correlation with the total metal concentrations. The relative fraction showed the highest percentage bioavailability for all metals in the reference sample, similar to the leaching test.

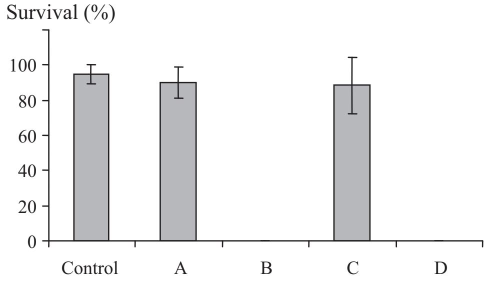

Fig. 3. The survival (mean ± SD) of *H. incongruens* in Ostracodtoxkit™ for the control soil sample and soil samples from sampling points A (reference), B*, C and D* after six days of exposure to the whole soil samples. *No surviving individuals in sample.

Soil leachates of sample A, C or D did not show any toxic effect in the Microtox® toxicity test, while sample B showed a slight toxic response.

The survival of the ostracods in the Ostracodtoxkit toxicity test was high for soil sample A and C, whereas the mortality was 100% for soil sample B and D, see Fig. 3.

Germination of radish was not affected by any of the soil samples B, C or D, in comparison with the reference sample A, see Fig. 4a. The germination of clover did, however, show a progressively negative trend for samples B, C and D, see Fig. 4b.

3.1. Determination of the risk with the Triad method

The risk of the soil samples was determined in line with the Triad method without any weighting of the different analyses according to their relevance. This indicated a relatively high environmental risk at the sampling points, see Table 1.

4. Discussion

Since both the Microtox® test and Ostracodtoxkit™ indicated an unexpected toxic effect of sample B and an ocular inspection

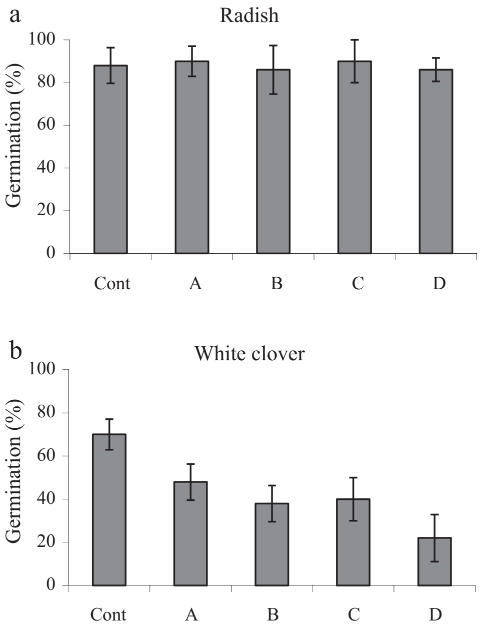

Fig. 4. (a and b) The germination of radish (70% germination according to manufacturer) and white clover (80% germination according to manufacturer) after exposure to a control soil (cont) and soil samples from the sampling points A, B, C and D.

Table 1
The scaled and integrated risks of the three soil samples B, C and D, (compared to reference soil sample A) determined according to the Triad method. The final risk value is determined from integrations of test results within and subsequently between each LoE.

	B	C	D
Scaled/combined risk			
Chemistry			
Total metals	0.23	0.77	0.91
Leaching tests	0.88	0.88	0.99
DGT	0	0	0.01
Ecotoxicology			
Ostracod toxicity test	1	0.02	1
Germination test$_{\text{(Radish)}}$	0.04	0	0.04
Germination test$_{\text{(Clover)}}$	0.21	0.17	0.54
Soil ecology			
C-mineralization + nitrification	0.78	0.80	0.75
Integrated risk (within the lines of evidence)			
Chemistry	0.55	0.70	0.95
Ecotoxicology	0.91	0.06	0.92
Soil ecology	0.78	0.80	0.75
Integrated risk (between the lines of evidence)			
Risk	0.79	0.62	0.90
Deviation	0.31	0.68	0.18

of the soil leachate raised suspicions of a petroleum contamination, the sample was analyzed for petroleum fractions (EN 14039; GC-FID analysis of oil index C10 to C40). The analysis showed total petroleum compound concentrations of 2370 mg/kg, which is assumed to be the cause of the toxic effect exerted by the sample. As the test organism in the Ostracodtoxkit$^{\text{TM}}$, *H. incongruens*, is a gill breather it is particlularly sensitive to compounds which may clog the gills. The oil detected in sample B was probably caused by a confined oil leakage at a parking lot on the site. A remediation of the oil found on the site is not justified as the site will continue to be used as a parking area.

The metal concentrations in sample B and C appear to have a negative effect on the C-mineralization. The relatively high C-mineralization at D was somewhat unexpected. The result could not be correlated to high metal concentrations or the nutrient status (C/N ratio).

The results from the nitrification experiments indicate an adverse effect of the metal contamination on the nitrification process of the indigenous bacteria at sampling point B and C. The toxic effect did not show any dose-response, the response was not greater in sample D than in B or C.

The findings from the germination test with radish were similar to those of Chapman et al. [25], who did not detect any toxic effect in the growth of wheat exposed to soil metal concentrations exceeding guideline values. This result highlights the necessity of including effect-based assessment methods in ecological risk assessments.

The final, calculated risk indicated an unacceptable environmental risk at all three sampling points. This should, however, not automatically lead to a direct decision of remediation though, with the exception of sampling point D. At this sampling point the calculated risk is high and the deviation in and between the different LoE is low, which suggests that the environmental risk is directly caused by the contaminant level. For sampling points B and C, however, the deviation is high. Therefore further ecotoxicological analyses, or alternatively a weighting of the analyses could provide data for a more reliable assessment. As the analyses in the ecotoxicological LoE give a direct indication of the toxic effect of the contaminants, this LoE should be given a higher weighting than the other two. Even within an LoE, some of the analyses performed may be weighted differently depending on their influence on the assessed risk of the sampling point. The high mortality in sample B in the ostracod test, for instance, indicated a high toxicity of the sample. This toxicity was, however, likely to be caused by a localised oil spill and not by metal contamination. As the oil spill is not any ground for remediation of the site, this test ought to be given a different weighting to reduce its relatively high impact on the integrated risk value.

4.1. Evaluation of the applicability of the Triad method

The Triad approach is useful for performing ecological risk assessments for many reasons; it is for example one of few quantitative WoE methods developed for assessing environmental risks [26]. As such, it is a transparent tool for risk assessment and evaluation, which lends itself well to the communication of risk between risk assessors and non-professional stakeholders [27]. Furthermore, it is a conceptually simple approach but the level of complexity of the actual risk assessment can be tailored to suit the requirements of the site investigation. In order to achieve a satisfactory risk assessment using the Triad method, producing reliable results, a breadth of different analyses are required. This obviously increases the demand of both financial and time resources. However, in comparison with the current Swedish ecological risk assessments, where the contaminant status at a contaminated site is often overestimated and incorrect decisions over remediation are made, these increased resources are relatively minor.

5. Conclusions

The risk assessment with the Triad method confirmed the environmental risk of sample D and showed that the toxicity of the sample was most likely caused by the high metal concentrations in the soil. The risk assessment of sample B and C was more complex, as there was a higher deviation between the results from the chemical analyses, ecotoxicological bioassays and the ecological inventory for these two samples. For sample B, a weighting of the results from the ecotoxicological LoE would be recommended in order to accurately determine the risk of the metal contamination at the sampling site as the toxic effect detected in the Microtox® test and Ostracodtoxkit$^{\text{TM}}$ test is more likely to be due to oil contamination. Sample C, on the other hand, requires further ecotoxicological testing, as the metal contamination levels are relatively high and the integrated risk value indicated an environmental risk from metal contamination.

In conclusion, the applied methodology, the Triad method, is considered appropriate for conducting improved ecological risk assessments in order to achieve sustainable remediation processes, which are more favorable from an environmental and societal perspective.

A development of more simple, cost-effective and standardized field analyses would improve the applicability of the Triad method in site-specific risk assessments.

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