



Applying multivariate analysis as decision tool for evaluating sediment-specific remediation strategies



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ABSTRACT

Multivariate methodology was employed for finding optimum remediation conditions for electro-dialytic remediation of harbour sediment from an Arctic location in Norway. The parts of the experimental domain in which both sediment- and technology-specific remediation objectives were met were identified. Objectives targeted were removal of the sediment-specific pollutants Cu and Pb, while minimising the effect on the sediment matrix by limiting the removal of naturally occurring metals while maintaining low energy consumption.

Two different cell designs for electrochemical remediation were tested and final concentrations of Cu and Pb were below background levels in large parts of the experimental domain when operating at low current densities (<0.12 mA/cm²). However, energy consumption, remediation times and the effect on naturally occurring metals were different for the 2- and 3-compartment cells.

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1. Introduction

Human activities have always affected the environment but with the industrial revolution, this has been accentuated and in contrast to early human activity, the problem reaches far beyond our local environment. Despite efforts to develop technology that will minimize pollution, there is still a need for treatment of already polluted water, soils, and sediments.

Sediments may contain complex mixtures of organic contaminants and/or heavy metals as pollution originates from different sources, e.g. shipping, shipyards, discharge of sewage, spills and from land-based activities. Depending on the nature of contamination and the location, it is sometimes possible to immobilize contaminants by capping. However, if sediments result from dredging, ex situ treatment will be necessary and to this end deep-sea dumping, aquatic containment and dumping at landfills have been employed. By treatment of sediments it is possible to minimize the amount of polluted material needing to be deposited (e.g. by removing coarser, less contaminated fractions) or even to be able to reuse the sediments for other purposes (e.g. as construction

material). The selection of the most appropriate technology for the remediation of sediments/soils thus depends on site-specific conditions, types of pollutants, pollutant concentrations and the end use of the treated sediment (Rulkens et al., 1998; Mulligan et al., 2001).

Organic pollutants are effectively destroyed by thermal remediation techniques but certain heavy metals (Hg, As and Cd) may be volatilized in the process and others (Mo and V) may become more leachable due to oxidation (Rulkens, 2005). Bioremediation (Guevara-Riba et al., 2004; Tabak et al., 2005) is one possible option, in which microorganisms are employed for immobilizing (White et al., 1997) or mobilizing (Erüst et al., 2013) metals that are part of natural biochemical cycles. Water soluble pollutants that are loosely bound to sediment particles may be solubilized before washing (Löser et al., 2007; Bing et al., 2008) and heavy metals have also been removed by extraction with strong acids (Dermont et al., 2008). Electrokinetic remediation offers another possibility that has been employed for both organic contaminants (Gomes et al., 2012; Méndez et al., 2012; Fan et al., 2016) and heavy metals (Gent et al., 2004; Kim et al., 2011; Iannelli et al., 2015).

Applying electro-dialysis, removal efficiencies of >90% of heavy metals have been achieved for waste materials such as soil, sediments, fly ash, wood and sewage sludge (Ottosen et al., 1995;

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Ribeiro et al., 2000; Ferreira et al., 2002; Jakobsen et al., 2004; Nystroem et al., 2005a) while maintaining low energy consumption (Sun and Ottosen, 2012). Electrodialysis is based on the principles of electrokinetic remediation and relies on an electric field of low current being applied to the polluted material. Ion-exchange membranes are employed to control the transport of ions to and from the polluted material. In this way acidification of the contaminated material causing mobilization of metals bound in the sediment is ensured by either water splitting at the anion exchange membrane (Ottosen et al., 2000) or by direct introduction of protons produced at the anode.

The need for developing tools for selecting the most appropriate remediation technology and identifying the optimal site-specific remediation conditions is apparent. For this, different statistical tools may prove valuable and in a recent report the use of factorial design in optimizing the remediation of uranium polluted soils was reported (Radu et al., 2015). Another method is projections onto latent structures (PLS) which has been extensively used in chemical synthesis (Carlson and Carlson, 2005a) and also in studies of the influence of soil properties on PAH oxidation (Jonsson et al., 2007) and for identifying the most relevant variables for electrochemical treatment of sediments (Pedersen et al., 2015b, 2015c, 2015d).

PLS is a method suited for handling large data sets by calculating the quantitative relationship between independent variables and responses. By arranging the observations (e.g. experimental settings, sediment characteristics) in an X-matrix and the responses (e.g. removal of contaminants, energy consumption) in a Y-matrix, the correlation may be calculated and optimal experimental settings, within the experimental domain studied, may be identified. In contrast to regression methods, PLS is based on projections, making it a robust method that tolerates missing data.

The focus of this study was demonstrating the applicability of PLS to a sediment-specific remediation strategy for harbour sediment, exemplified by electro-dialytic remediation. PLS was employed to determine optimal experimental settings in regard to the remediation objectives and was also used to evaluate energy consumption in the different parts of the experimental domain.

2. Methods and materials

2.1. Sediment analyses

The sediment from Hammerfest harbour (40.17°N, 41.32°E), located in Norway, used in this study, has previously been found to contain levels of Cu and Pb above background levels according to the Norwegian sediment quality criteria (Pedersen et al., 2015a).

The sediment was from the top 10 cm of the seabed using a Van Veen grab in Hammerfest harbour, Norway. The samples were kept cool during transport and stored in a freezer (−18 °C) until analysed or treated.

Major elements and heavy metal concentrations (Al, Fe, K, Mg, Mn, As, Cr, Cu, Ni, Pb, Zn) were measured based on digestion (Norwegian standard NS4770). Sediment dried at 105 °C (1.0 g) and HNO₃ (9 M, 20 mL) were autoclaved (200 kPa, 120 °C, 30 min). Solid particles were subsequently removed by vacuum filtration through a 0.45 µm filter and the liquid was diluted to 100 mL. Metal concentrations in the liquid were measured by Inductively Coupled Plasma - Optical Emission Spectrometry (ICP-OES).

Carbonate content was measured by treating dried sediment (5.0 g) with HCl (3 M; 20 mL) and the developed CO₂ was measured volumetrically in a Scheibler apparatus, calibrated with CaCO₃.

Organic content was based on loss of ignition on dried sediment (2.5 g) being heated at 550 °C for an hour.

pH (KCl). Dried sediment (5.0 g) was agitated with KCl (1 M, 12.5 mL) for an hour and pH was subsequently measured using a

radiometric analytical electrode.

Grain size distribution was measured by wet sieving and dry sieving. Wet sediment (75 g), distilled water (350 mL) and Na₄P₂O₇·10H₂O (0.1 M, 10 mL) was agitated for 24 h. The slurry was then sieved through a 63 µm sieve and the fraction above 63 µm was subsequently dried and sieved for 15 min in a mechanical shaker using sieves with screen openings of 0.063, 0.080, 0.125, 0.25, 1.0 and 2.0 mm. The slurry fraction below 63 µm was transferred to Andreason pipette for gravitational sedimentation. Stoke's law was used for measuring time required for particles to settle 20 cm and samples representing the sizes 40, 32, 16, 8, 4, 2 and 1 µm were sampled.

Sequential extraction was made in four steps based on the improvement of the three-step method (Rauret et al., 1999) described by Standards, Measurements and Testing Program of the European Union. Air-dried sediment (0.5 g) was first extracted with acetic acid (0.11 M, 20 mL, pH3) for 16 h; secondly extracted with hydroxylammonium chloride (0.1 M, 20 mL; pH2) for 16 h; thirdly extracted with hydrogen peroxide (8.8 M, 5 mL) for 1 h, followed by extraction at 85 °C for 1 h, followed by evaporation of liquid at 85 °C, subsequently the cooled solid fraction was extracted with ammonium acetate (1 M, 25 mL, pH2) for 16 h; and fourthly digestion on the remaining solid particles was made, following the description above.

2.2. Electro-dialytic remediation experiments

2.2.1. Materials and procedure

Two different electro-dialytic remediation (EDR) cells were employed in the study. The set-up and principles of the cell designs are illustrated in Fig. 1. The difference between the two is the insertion of an anode compartment in the extensively used 3-compartment cell. Acidification has been shown to occur faster in the 2-compartment cell and with a lower energy consumption. The faster mobilisation of metals may result in relatively higher release of naturally occurring metals into the liquid phase and for this reason the 3-compartment cell has in this study been used as a reference. Stirring has proven more efficient than a stationary set-up (Pedersen et al., 2003; Ottosen et al., 2012) and appears appropriate to apply for remediating dredged sediments. Other variables of importance to the efficiency of EDR include sediment, cell design, current density and time (Pedersen et al., 2015d, 2015e).

The 2-compartment and 3-compartment cells used in the study were designed using the same materials and sizes; the 3-compartment cells consisted of two electrolyte compartment and between these a compartment containing the sediment suspension. The 2-compartment cell consisted of one electrolyte compartment (catholyte) and a compartment containing the sediment suspension. The cell compartments were manufactured from Plexiglas and the dimensions were: length of electrolyte compartments 3.5 cm; length of sediment suspension compartment 10 cm; inner diameter of all compartments 8 cm. Ion exchange membranes from Ionics (anion exchange membrane 204 SZRA B02249C and cation exchange membrane CR67 HUY N12116B). The electrolyte was NaNO₃ (0.01 M) adjusted to pH 2 by HNO₃ (5 M). The electrolyte liquids (350 mL) were circulated via Pan World pumps with flow rates of 30 mL/min. Platinum coated titanium electrodes were used in each electrolyte compartment and a power supply (Hewlett Packard E3612A) maintained a constant DC current. The sediment suspension was stirred by a RW11 Basic lab-egg (IKA 2830001) with a stirrer consisting of plastic flaps (4 cm × 0.5 cm) fastened to a glass rod.

After the EDR experiments the sediment suspensions were filtered and the heavy metal concentration in both the suspension liquid and solids were measured. The stirrer, membranes and

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