



CHARACTERISATION OF DREDGED MATERIALS

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ABSTRACT

Management of contaminated sediments requires - similar to other waste materials - a holistic approach. In addition to the common predictive techniques for estimating contaminant losses - *a priori* techniques and pathway-specific tests - the interactive nature of various parameters has to be recognized. This means, that particular emphasis should be placed on the evaluation of the two data sets "driving force/geochemical gradient" parameters (mostly redox-sensitive components, which may be critical for the "acid producing capacity") and "capacity controlling properties" (with prevalence of "carbonate content"). Assessment of these parameters and their long-term borderline conditions, as well as the potential links between the two sets of geochemical properties and of individual parameters should be an integrated part of the wider management scheme, i.e., the analytical and experimental parameters should always be related to the potential remediation options for a specific sediment problem. © 1998 Published by Elsevier Science Ltd on behalf of the IAWQ. All rights reserved

KEYWORDS

Capacities; quality criteria; disposal; predictive techniques; resuspension.

INTRODUCTION

Dredging is a human activity, which can well compete with highly dynamic natural sedimentary processes such as landslides. In the 1980s, the International Association of Ports and Harbors estimated about 350 million tonnes of maintenance dredging and 230 million tonnes average annual new construction dredging. In the harbours around the North Sea, approximately 100 million m³ of sediment has to be dredged annually - about 10 times the average annual sediment discharge of Rhine River. Typical problems with these sediments are:

- increasing volumes of dredged materials,
- high concentrations of toxic substances,

and these problems have been concentrated mainly at the mouth of large rivers and in coastal areas.

Due to the economic implications, there is increasing world-wide interest in the development of dredging and disposal technologies. Among the authorities particularly dealing with the subject of contaminants in dredged materials the U.S. Army Corps of Engineer Waterways Experiment Station at Vicksburg, Mississippi, has played a leading role. In the early eighties, the Environmental Laboratory of this institution

together with U.S. Environmental Protection Agency initiated a "Decision-Making Framework for Management of Dredged Material Disposal", which includes test procedures on physicochemical conditions, aquatic bioaccumulation, and water column effects both at the site of dredging operations and disposal of dredged materials. Further intensification of coordinated research was performed by the ARCS ("Assessment and Remediation of Contaminated Sediments") group of USEPA, which has been focussing on the Great Lakes Areas of Concern (1990-93); an "Integrated Contaminated Sediments Assessment Approach" (Anonymous, 1994) includes six topics "sampling design and quality control", "sample collection", "chemical analysis", "toxicity testing", "benthic community structure and survey", and "tumors and abnormalities". In addition, the ARCS Program was charged with assessing and demonstrating remedial options for contaminated sediment problems in the Great Lakes; laboratory tests were conducted utilizing 13 processes, and pilot-scale (field-based) demonstration of bioremediation, particles size separation, solvent extraction and low temperature thermal desorption were conducted (Averett, 1993).

It seems, that sufficient data have been assembled by these and other organizations with respect to both risk assessment methods and remediation procedures for contaminated sediments which should allow development of more integrative perspectives in this subject. The present geochemical approach is emphasizing the interactive nature of chemical parameters and is focussing on the long-term effects of pollutant release from disposed sediments.

RISK ASSESSMENT OF CONTAMINATED SEDIMENTS

Various strategies have been proposed during the last ten years for decision-making on contaminated sediment. One of the earlier schemes of USEPA (1979) following identification of problem areas and critical chemicals first decides on priority sources. With ongoing sources, the maximum percentage of possible source control is estimated, as well as the question, if recovery can be accomplished in an acceptable timeframe. If the question on ongoing sources is denied, an evaluation takes place on combined sediment remedial action and source control. If the sources have been stopped and recovery cannot be expected in acceptable time frame, then sediment remedial action has to be evaluated.

Beside the costs of the remediation technique, the major questions relate to the contaminant loss pathways. Contaminant loss can occur through one or more pathways. The example of a confined disposal facility indicates, that the potential pathways for contaminant loss include surface runoff, effluent, seepage, leachate, dust and uptake by plants and animals. Predictive techniques for estimating contaminant losses comprise two categories (Myers *et al.*, 1993):

- *a priori* techniques which are suitable for planning-level assessments, and
- techniques that use pathway-specific test data provide state-of-the-art loss estimates (generally more advanced techniques).

For some remediation components there are no pathway-specific tests and *a priori* techniques for all pathways of all components available. Confidence and accuracy for *a priori* loss estimates from confined disposal facilities are low. For test-based loss estimates they vary with the stage of development of the test. Confidence is high for test-based estimates of leachate losses. Confidence and accuracy are high for estimation of test-based runoff loss.

Typical methods for measuring physical and engineering properties of contaminated sediments - *a priori* techniques - as recommended in the early seventies comprise: particle size distribution, organic content - measured as total volatile solids - solids content, liquid and plastic limit test, void ratio and density.

SEDIMENT QUALITY CRITERIA

During the last 20 years, considerable experience has been assembled with pathway-specific test data, in particular with more innovative treatment procedures. However, most important progress in risk assessment

of contaminated sediments, has been made in the context of sediment quality criteria development, following the experience, that long-term perspectives in water management need "integrated strategies", in which sediment-associated pollutants have to be considered as well. In particular, it has been shown, that for most cases of surface waters, the contaminant level in the sediment has greater impact on the survival of benthic organisms than do aqueous concentrations.

First efforts have been undertaken by the United States Environmental Protection Agency to develop standard procedures for the assessment of environmental impact of sediment-bound pollutants. Further discussions led to the differentiation of biological and chemical-numerical approaches. Biological criteria integrate sediment characteristics and pollutant loads, but they do generally not indicate the cause of effects. With respect to chemical-numerical criteria, there is no immediate indication on biological effects; their major advantages lie in their easy application and amendability to modeling approaches.

Biological criteria

Biological approaches on development and application of sediment quality criteria exhibit a common basis in the study of damaging impacts from contaminated sediments on organisms. The biological parameters "bioaccumulation", "toxicity", and "mutagenity" have to be considered separately in any case. Bioassays as well as field surveys are empirical considerations which cannot provide numerical criteria to be transferred onto different situations.

Selection of the optimal methods for assessing aquatic ecosystem degradation and potential risks from contaminated sediments will depend on the study objectives, resources, and the characterization of the methods; this is particularly valid for biological test procedures, which increasingly form the key aspect in an integrated approach, characterizing the physicochemical conditions (including habitat and contaminant patterns), indigenous biological communities, and toxicity (Burton, 1993).

Available biological sediment quality criteria have been checked, if they respect the requirements to be generally applicable (to different sites and all exposition pathways) and comprehensive (to different trophic levels and a wide spectrum of contaminants). According to the present experience, "bioassay approach" and "sediment quality triad" are closest to these requirements. The sediment quality triad (Chapman, 1986) is an integrated procedure, which uses empirical evidence, that is observation, not being based on theories; such procedures seem to be particularly promising, since each component of the system is contributing to the interpretation of the other components. The "triad approach", although not standardized at present, may nonetheless lead to general sediment quality criteria, however, at relatively high costs.

Relatively simple and implementable liquid, suspended particulate and solid-phase bioassays have been carried out for assessing the short-term impact of dredging and disposal operations on aquatic organisms (Ahlf and Munawar 1988). Standardized tests are characterized by their lack of variability, but essential information (e.g., lethality, alterations of growth rate) can only be obtained with such single species test. The influence of the main environmental variables on the interaction of suspended particulates or in-situ sediment contaminants and organisms should also be determined under simulated field conditions. In particular, benthic bioassay procedures, due to recent developments, are important in evaluating the relationship between laboratory and field impacts (Reynoldson, 1987).

The concept of Ahlf and collaborators (1992) for the assessment of sediment-bound pollutants is mainly based on microbial toxicity tests, using bacteria and algae. An overall biological assessment scheme includes:

- field description of benthic communities,
- benthos bioassay on total sediment,
- porewater bioassay (bacteria, Chromotest),
- elutriate bioassay (algae, bacteria, Chromotest).

In addition, tests can be performed on fractions of the sediment, which have been extracted or treated with a cosolvent. For example, a non-polar surfactant has been applied, which is commercially used to solubilize hydrocarbons from contaminated soil. On the other hand, the concept can be modified according to users requests, in that only parts of this structure may be needed for a site-specific problem.

In practice, the following questions have been posed for test procedures - in particular biotests - on contaminated dredged sediments:

- What are the most toxic sites?
- Where should remediation start?
- What technique has to be used, in accordance with the site-specific pollutant load?
- Is there a difference in the toxicity of sediments, which are resuspended into the water phase and those, which are buried in deeper layers?
- What is the minimum number of assays for non-redundant information?
- What is the relation between different assays and chemical data?

The latter two questions are closely related to the costs of such investigations. From the present experience, including a recent evaluation of about one thousand literature references, we come to the following conclusions (Förstner *et al.*, 1994): as far as equilibrium conditions can be assumed for sorption and desorption processes, one should restrict toxicological investigations to the water phase. Biological effects, which are suspected to be caused by sediment-bound contaminants should preferably be assessed by laboratory experiments. On the other hand, if the effect of "in place" pollutants on the abundance and distribution of benthic organisms have to be studied, this should be done from field data

We should emphasize the merits of microbial bioassays, which are rapid, inexpensive and sensitive to toxicants. In many cases microbiotests are adequate substitutes for expensive macro-biotests. We can conclude that the tools for biological investigations are basically available, especially in the area of bioassays. What is still missing, are the agreements on standardized concepts and procedures.

Open questions relate to the implications of anoxic sediments, mainly with respect to the effect of toxic metals. In a sulfidic anoxic environment, even if the sediment is heavily polluted by metals, organisms are still considered to be safe due to the strong fixation of metal ions by S²⁻ or HS⁻ as source of acid volatile sulfide. Such heavily polluted sediment, however, can behave as a time bomb which is triggered by only one factor - redox increase to a critical point, i.e., by exposure to oxygen-rich overlying water or directly to air. Once this situation occurs (oxygen transfer via plant roots is also a possible pathway), toxic metals in the sediments will be released to the water phase or transformed into more bioavailable species.

Chemical numerical criteria

Numerical approaches for the assessment of environmental impact of sediment-associated metals are based on:

- accumulation,
- pore water concentrations,
- solid/liquid equilibrium partition (both sediment/water and organism/water),
- elution properties of contaminants.

Background Approach: Compares the actual data with sites comprising natural or insignificant pollutant concentrations. Particularly useful are samples from deeper layers of the sediment sequence at a given site, for example, from drill holes, since this material is derived from the same catchment area and usually is similar in its substrate composition. None the less, standardization with respect to grain size distribution is indispensable.

Porewater Approach: Based on the experience that the composition of interstitial waters is the most sensitive indicator of the reactions that take place between pollutants on particles and the aqueous phase which contacts them. There is the advantage of a direct recovery and analysis of water-borne constituents. But there are several disadvantages, mainly arising from the sampling and sample preparation, which need considerable precautions, such as for exclusion of oxygen.

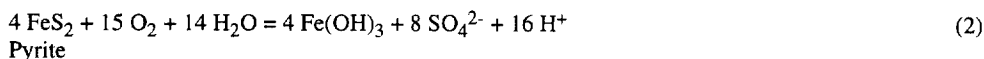
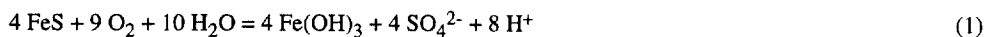
Equilibrium Approaches: These approaches are related to the broad toxicological basis of food and water quality data - a very important advantage. On the other hand, there are the effects of sample preparation, e.g., the drying procedures; the separation techniques, for example, filtration or centrifugation; there are strong effects of grain size composition and the influence of suspended matter concentration in the aquatic system, which is even more important, if the kinetics of sorption and desorption are too slow for equilibrium to be achieved in a given time of interactions. Unlike non-ionic organic chemicals, K_D -values of metals are not only correlated to organic substances but also with other sorption-active surfaces; therefore, the equilibrium partition approach exhibits strong limitations for metallic elements.

Remobilization: Short-term effects may be studied from water/sediment-suspensions, medium-term effects from experiments using tanks, and long-term effects by applying chemical extractants, either singly or in sequence. Field observations often do not show clear effects, as has been demonstrated for the release of metals from anoxic sediments during oxidation. Such implications for future criteria development, particularly important for dredging and management of dredged material, will be discussed on the basis of experience from metal speciation studies on soils and sediments.

LONG-TERM EFFECTS, PARTICULARLY OF REDOX PROCESSES

Reliability of long-term prognoses on the impact of metal-contaminated sediments is particularly restricted due to the dominance of non-linear and delayed processes in redox- and pH-controlled systems. Acidity is perhaps the most serious long-term threat from metal-bearing wastes. For decades, water seeping from mine refuse has delivered increased metal concentrations into receiving waters. The threat is especially great in waters with little buffer capacity. The acidity production can develop many years after disposal, once the neutralizing or buffering capacity in a pyrite-containing waste is exceeded.

The major process (see equations 1 to 3) affecting the lowering of pH-values (pH 2 to 3) is the exposure of pyrite (FeS_2) and other sulphide minerals to atmospheric oxygen and moisture, whereby the sulphidic component is oxidized to sulphate and acidity (H^+ ions) is generated. Bacterial action can assist the oxidation of $\text{Fe}^{2+}(\text{aq})$ in the presence of dissolved oxygen.



The acidification of a sediment/water system begins after hydrogen ions are generated during the oxidation (e.g., during dredging or resuspension of mainly fine grained material containing less carbonate than needed for long-term neutralization). Primary emissions containing high metal concentration issue from waste rocks and tailings, while tailing ponds are primarily responsible for secondary effects on groundwater. An important and long-term source of metals are the sediments reworked from the flood plain, mainly by repeated oxidation and reduction processes.

High concentration factors were found in inland waters affected by acidic mine effluents. The concept of acid-producing potential (APP) was initially developed in the prediction and calculation of acid mine drainage and waste tailings management (Anonymous [1979]; as summarized by Ferguson and Erickson [1988]). Our findings on the effects of periodical redox processes on both APP and metal mobility in

estuarine sediments (Kersten *et al.*, 1985; Kersten and Förstner, 1991) have further enhanced research interest in this field. Periodical redox processes can spur an increase or decrease in APP or pH in a sediment/water system. In a closed system, periodical redox processes can lead to the change or transfer between APP(s) and APP(aq) but the total APP of the system does not change. The processes are reversible. The hydrogen ions produced in the oxidation will be consumed by the following reduction. Contrarily, in an open system, the total APP of the system will change depending on the properties of the system and the reaction processes. Under certain conditions total APP in the system increases, while under other conditions total APP in the system decreases. Some processes are irreversible. The components producing or consuming H^+ leave the system and cause the change in APP(s), APP(aq) and permanent ANC (acid neutralizing capacity); this is the result of, for example, "split of sulfate" (Van Breemen, 1987).

Direct assessment of the pH-changes resulting from the oxidation of anoxic sediment constituents can be performed by ventilation of sediment suspensions with air or oxygen and subsequent determination of the pH-difference between the original sample and oxidized material. The greater is this difference, the higher is the short-term mobilization potential of metals, e.g. during dredging, resuspension and other processes, by which anoxic sediments come into contact with oxygenated water or - following land deposition of dredged material - with atmospheric oxygen.

Pore water data from dredged material from Hamburg Harbor indicate typical differences in the kinetics of proton release from organic and sulphidic sources. Recent deposits are characterized by low concentrations of nitrate, cadmium and zinc; when these low-buffered sediments are oxidized during a time period of a few months to years, the concentrations of ammonia and iron in the pore water typically decrease, whereas those of cadmium and zinc increase (with the result that these metals are easily transferred into agricultural crops!).

Of the two major release processes, the first - oxidation of sulphides - can be predicted to some extent, whereas the implications of long-term degradation of organic matter on the release of less mobile elements such as copper and lead as yet cannot satisfactorily be described. Here, as with the effects of these interactions on the cycles of anionic elements such as arsenic, further research is needed.

Experimental approaches for calculating APC and ACC for sulfidic mining residues have been summarized by Ferguson and Erickson (1988). A test described by Sobek *et al.* (1978) involves the analysis of total pyritic sulfur; potential acidity is then subtracted from neutralizing potential which can be obtained by adding a known amount of HCl, heating the sample and titrating with standardized NaOH to pH 7. Bruynesteyn and Hackl (1984) calculated APC from total sulfur analysis; here, acid-producing capacity was then subtracted from acid-consuming capacity, obtained by titration with standardized sulfuric acid to pH 3.5. The APC-relationships of sediments are more complex than that in sulfidic ores because the APC from organic matter must be considered; time scale plays a major role in sediments.

The most efficient fixation process within anoxic sediments for trace metals is production of free sulfide during anaerobic organic matter degradation and reduction of sulfate (study of heavy metal associations with sulfide and carbonate formations in anoxic sediments, therefore, provides insight into early diagenetic processes; Berner, 1981, Morse and Mackenzie, 1990).

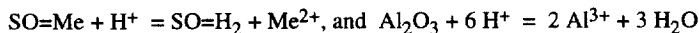
Whereas the ability of the sediment to produce free sulfide is determined by the sulfate reduction rates, the ability to remove all produced free HS^- is given by the reactive metal - predominantly reducible Fe^{3+} concentrations - available to form sulfide minerals ("Available Sulfide Capacity" [ASC]; Williamson and Bella, 1980). Simultaneous application of standard sequential leaching techniques (BCR-Version: Ure *et al.*, 1993) on critical trace elements and matrix components can be used for geochemical characterization of anoxic, sulfide-bearing sediments in relation to the potential mobility of critical trace metals (Kersten and Förstner, 1991). For determining the acid producing capacity ("maximum APC") in anoxic sediments, both the FeS pool (actual APC) and the maximum ferrous sulfide (worst case: pyrite)-producing capacity upon disposal has to be taken into consideration.

ASSESSING LONG-TERM MOBILITY OF METALS IN SEDIMENTS BY TITRATION EXPERIMENTS

With regard to prediction of long-term effects of sediment-bound metals, chemical extraction procedures are of limited value because they usually involve neither reaction-mechanistic nor kinetic considerations. This lack can be avoided, e.g., by an experimental approach, originally been used by Patrick *et al.* (1973) and Herms and Brümmer (1978), where sediments can be treated in a circulation system under controlled intensification of significant release parameters such as pH-value, redox-potential, and temperature. Our experimental design (Schoer and Förstner, 1989) includes an ion-exchange system for extracting and analyzing the released metals at an adequate frequency, and compares sequential extraction results before and after treatment of the sample in the circulation apparatus. Individual metal species are released at different time intervals. Taking into account both element contents released during the 10 weeks experiments (equivalent to several thousand years of solid/water interaction) and those extrapolated from extraction "pools", concentrations can be calculated for different scenarios.

While these extrapolations have been made from pH 5 conditions, titration curves from investigations on a wide spectrum of metal-bearing waste materials (Obermann and Cremer, 1992) suggest that pH 4 may be more appropriate for long-term predictions of potential metal releases from contaminated sediments. In this pragmatic approach, the pH is automatically adjusted to 4 during the time period of 24 hours; apart from the release rates of metals, which can be determined from samples taken at different time intervals, the sum curve (diagram of discrete acid additions would be valuable!) of acid consumption provides informations on the potential changes of the matrix composition during acidification and the availability of buffer capacity at different time scales.

Acid consumption reflects slow long-term metal release from sediments. Because calcite dissolution was fast, the acid consumption in the first stage increased drastically within a short period of time. Cation release and aluminosilicate dissolution were dominant factors consuming acid in the later stage. The reactions can be treated as:



where SO= and Me are reaction groups in solid phase and metal ion, respectively. The reactions of hydrogen ion with metal and aluminosilicate are delayed due to the complex sediment structure and matrix. For example, special penetration of hydrogen ion is required for reacting with cation on clay minerals coated with organic matter or biofilms. Rates of reactions can be estimated by measurements of metal concentrations in solution.

In dredged material management, two different target areas for combined matrix/metal criteria can be distinguished:

- sediment resuspension
- dredged material disposal

With respect to "resuspension of aquatic sediments", which involves more short-term effects than the disposal of dredged material, special emphasis should be placed on the factor "available metal species". Within certain categories of acid producing and consuming capacities, guideline values for individual metals should be based on elutriate data, preferentially at pH 4, for better comparison with other solid matrices (e.g., Swiss Ordinance for Waste Materials [Anonymous, 1990]). Environmental impact of sediment deposits is influenced by the internal chemical conditions rather than by the concentration and extractability of metals. Priority, therefore, should be given to the optimization of long-term chemical stability ("geochemical engineering").

At the moment, research on long-term effects of redox variations on metal behaviour in sediments is mostly based on thermodynamic considerations. Future research should emphasize studies on the kinetics of metal

species transformations, hydrogen ion production and metal release as affected by changing redox conditions. Additional important aspects involve the bridging of the gaps between numerical criteria approaches, as reflected by matrix composition and metal mobility, and biological approaches. It may well be, that for such systems, which are much less disturbed than artificial sediment elutriates, relationships between matrix conditions - as reflected, e.g., by redox indices - and metal species bioavailability may be found, which may serve as a more solid basis for the interpretation of results from bioassays, eventually with respect to chronic toxicity.

CONCLUSIONS

Technological options are more restricted for dredged sediments than for other waste materials in most cases. In particular, remediation techniques in the closer sense are often economically unacceptable because of the large volume of contaminated sediments. The widely diverse contamination sources in larger catchment areas usually produces a mixture of pollutants, which is more difficult to treat than a specific industrial waste. Even if one has procedures at hand to reduce 9 priority pollutants below the guideline values, number 10, for example for mercury or PCB may render the whole business unrealistic. There is a long retention time for sediments in larger catchment areas. Improvement at the source may need decades to become effective in the sediments at the lower reaches and harbours close to the river mouth. For most sediments from maintenance dredging, there are more arguments in favor of "disposal" rather than "treatment". Final storage conditions would imply, that these materials should be deposited in a favourable chemical environment. At the actual state of knowledge, this could only mean "permanent" anoxic conditions". Such conditions can be made artificially or be selected from natural environments. However, even for these stable geochemical conditions, which are provided, for example, in natural environments such as the Black Sea and fjords, not all potential implications for long-term pollutant release and transformations are known, and therefore, further research is needed.

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