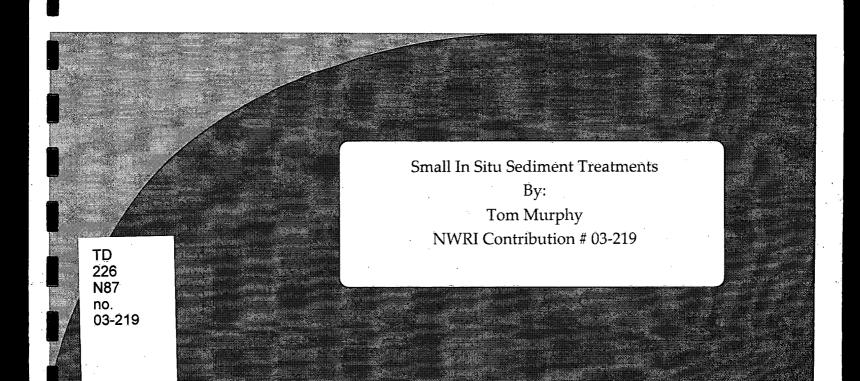
Environment Canada

Water Science and Technology Directorate

Direction générale des sciences et de la technologie, eau Environnement Canada



Small In Situ Sediment Treatments

Tom Murphy

Abstract

Prior to full-scale sediment treatment, it is necessary to conduct small-scale treatment evaluations. In situ assessments have some advantages over laboratory treatments with respect to maintaining relevant temperature, redox, and light conditions. Direct injection with divers allows for in situ incubations but the lack of control over the treatment produces variance that makes optimization of the treatment difficult. To have the advantages of both in situ and laboratory approaches, we collected box cores via a diver, mixed in the reagents at the surface and put the samples back into the hole in the sediments where they came from. Evaluations of equipment for full-scale remediation are also required to optimize the treatment process.

NWRI RESEARCH SUMMARY

Plain language title Small In Situ Sediment Treatments

What is the problem and what do sicentists already know about it?

Sediment remediation is considered for many contaminated sites but the tools for evaluations were not well established. In situ bioremediation is considered as an alternative to sediment dredging and subsequent treatment. There are many limitations including the engineering of injection of the treatment chemicals. Small-scale protocols to optimize treatments are required.

Why did NWRI do this study?

NWRI was asked to evaluate sediment treatment in the St. Marys River as part of the remedial action process. NWRI also tested sediment injection equipment to initiate commercial treatment of contaminated sediments.

What were the results?

NWRI demonstrated ten years ago that sediment remediation was technically possible. The equipment was successfully used later in other sites to effectively remediate contaminated sediments. We discovered that the contaminated sediments at the St. Marys River were much deeper than had been anticipated. The coal tar and wood fibre were as deep as two meters. The ability to produce a bioremediated cap over such a deposit remains unresolved anywhere. The small scale apparatus for evaluating in situ sediment treatment is highly effective.

How will these results be used?

Full-scale commercial treatment of sediments is proceeding under a license agreement in Hong Kong and elsewhere. The tools for testing sediment remediation are also used by business partners in Asia and USA. The associated presentation in Ho Chi Minh City reviewed the concepts of in situ sediment treatment and the paper provides a basic background.

Who were our main partners in the study?
The Great Lakes Cleanup Fund and the Biotechnology Fund supported this project.

Traitement in situ à petite échelle de sédiments

Tom Murphy

Résumé

Avant d'effectuer le traitement de sédiments en grandeur réelle, il faut évaluer des interventions à petite échelle. À cet égard, les études in situ présentent certains avantages par rapport aux évaluations en laboratoire quant au maintien des conditions de température, redox et de luminosité adéquates. L'injection directe de produits par des plongeurs permet l'incubation in situ, mais il est difficile d'optimiser le traitement, car le manque de contrôle des conditions entraîne des variations. Pour réunir les avantages des méthodes de traitement tant in situ qu'en laboratoire, nous avons prélevé des échantillons au moyen d'un carottier à boîte manipulé par un plongeur, mélangé les matières prélevées avec les réactifs à la surface et réintroduit les mélanges dans les sédiments là où les échantillons avaient été prélevés. Il faut également évaluer l'équipement nécessaire aux opérations de restauration à grande échelle pour optimiser la méthode de traitement des sédiments.

Sommaire des recherches de l'INRE

Titre en langage clair

Traitement in situ à petite échelle de sédiments

Quel est le problème et que savent les chercheurs à ce sujet?

On envisage l'assainissement de sédiments en maints endroits contaminés mais, jusqu'à maintenant, les outils d'évaluation des interventions n'étaient pas au point. La biorestauration in situ de sédiments est considérée comme une solution de rechange à l'enlèvement par dragage et au traitement ultérieur. Cette approche comporte cependant de nombreuses limitations, dont les aspects techniques de l'injection des produits chimiques servant au traitement. Il est donc indiqué d'appliquer des protocoles d'évaluation à petite échelle pour optimiser le traitement en grandeur réelle.

Pourquoi l'INRE a-t-il effectué cette étude?

On a demandé à l'INRE d'évaluer le traitement des sédiments dans la rivière St. Marys dans le cadre des opérations de restauration. L'INRE a également fait l'essai de l'équipement d'injection des produits chimiques en vue d'amorcer le traitement commercial de sédiments contaminés.

Quels sont les résultats?

L'INRE a démontré il y a dix ans que la restauration de sédiments contaminés est techniquement réalisable. On a réussi plus tard à décontaminer des sédiments avec l'équipement utilisé à cette fin. Nous avons constaté que la couche de sédiments contaminés de la rivières St. Marys était beaucoup plus épaisse que prévu : les

accumulations de goudron de houille et de fibre de bois pouvaient atteindre jusqu'à deux mètres d'épaisseur. Nulle part n'a-t-on encore pu décontaminer une strate de sédiments par biorestauration dans la partie supérieure de dépôts de cette envergure. L'appareil utilisé pour l'évaluation in situ à petite échelle du traitement de sédiments est très efficace.

Comment ces résultats seront-ils utilisés?

On effectue des traitements en grandeur réelle sur une base commerciale au titre d'un contrat de licence à Hong Kong et ailleurs dans le monde. En outre, des partenaires commerciaux utilisent les outils d'évaluation de la décontamination de sédiments en Asie et aux États-Unis. La présentation connexe faite à Hô Chi Minh-Ville (anciennement Saigon) passait en revue les notions du traitement in situ des sédiments. L'article fournit de l'information générale à ce sujet.

Quels étaient nos principaux partenaires dans cette étude?

Le soutien financier du projet a été assuré par le Fonds d'assainissement des Grands Lacs et le Fonds des biotechnologies.

Small In Situ Sediment Treatments

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Abstract

Prior to full-scale sediment treatment it is necessary to conduct small-scale treatment evaluations. In situ assessments have some advantages over laboratory treatments with respect to control over temperature, redox, and light. Direct injection with divers allows for in situ incubations but the lack of control over the treatment produced variance that makes optimization of the treatment difficult. To have the advantages of both in situ and laboratory approaches, we collected box cores via a diver, mixed in the reagents at the surface and put the samples back into the hole in the sediments where they came from. Evaluations of equipment for full-scale remediation are also required to optimize the treatment process.

Introduction

Most sediment treatment has been associated with sediment dredging and ex situ confinement or treatment. At times, it is more cost-effective or less intrusive to treatment contaminated sediments in situ. In situ sediment treatment is still a relatively new concept in that few sites have been restored with this approach. In situ treatments have used geotechnical or biological concepts but at times the distinctions are blurred.

Geotechnical Sediment Treatment

Most capping of contaminated sediments has used relatively inert sand. Because of the lack of reactivity of sand and need to block availability and accessibility of the contaminant, the sand layer is generally 0.5 m or more thick. Recently more reactive materials like zero valent iron, coke or apatite have been used to provide a reactive cap (Lowry 2003, Melton et al. 2003, Reible et al. 2003, Wiesner et al. 2003). The choice of reactive materials depends upon the contaminant. Zero valent iron is capable of enhancing bioremediation of organic contaminants whereas some reactive materials are effective at metal complexation. The ability to place a layer of reactive materials over contaminated sediments is largely an engineering exercise but the design of the reactive materials involves many disciplines including geotechnical, geochemical, microbial and biological.

Most applied limnological treatments of mercury have used selenium to inactivate mercury (Hültberg et al. 1999, Kukkonen and Nuutinen 1999), but there are concerns about the skill required to titrate one toxin with selenium, another toxin. Naturally, the distribution of mercury has been observed to adsorb to iron (Quemerais et al. 1998, Ribeiro et al. 1999) but iron is not used for mercury treatment. Iron has been added to lakes and sediments to precipitate phosphorus and reduce eutrophication. NWRI has handled iron in both pilot-scale sediment (Murphy et al. 1995) and full-scale lakewater treatment (Hall et al. 1994). Iron is reactive and will seal the existing sediments by dimerization and other bridging reactions so the geotechnical aspects should be

considered as well. In sediment treatments with calcium nitrate we observed long-term physical stabilization of sediments (Amos et al. 2003)

Bioremediation

Many studies have shown that simple addition of inorganic fertilizers stimulated bioremediation of freshly spilled oil (Lee and Mora 1999, Lin et al. 1999). In samples from Iran, Alemzadeh and Vossoughi found both inorganic and organic forms of nitrogen limited oil bioremediation. Jackson and Pardue (1999) found that nitrogen as ammonium (100-670 mg/L) was most effective amendment for stimulation of oil in Louisiana marine marshes and that phosphorus was not limiting. But at other sites, phosphorus can also limit bioremediation (Wright et al. 1997). Sometimes both phosphate and nitrogen have been shown to stimulate biodegradation (Labare and Alexander 1995). Durant et al. (1997) have shown that addition of oxidants can stimulate bioremediation of aromatic hydrocarbons, but the bioremediation response to oxygenation varies considerably within an aquifer. Both Durant et al (1997) and Robertson and Alexander (1992) proposed that phosphate precipitation was the likely mechanism of suppression of microbial activity and our ongoing studies of mineral precipitation in Kuwait sediments indicate that P-precipitation may be a problem. At times, oleophilic fertilizers are used in part to overcome this precipitation of phosphorus (Churchill et al. 1995).

Efforts to enhance rates of bioremediation by adding inoculums of microbes have generally failed in large treatments (Atlas 1995, Lee 1999, Lin et al. 1999). At times, special formulations such as oleophilic fertilizers have been used to enhance bioremediation of oil (Atlas 1995). Iniplo EAP22 is an oleophilic fertilizer composed of a micro-emulsion with urea, laureth phoshate and oleic acid. Lee (1999) reviewed how Inipol EAP22 failed in lab and field demonstrations to stimulate bioremediation. The failures were mainly associated with washout of the nutrients. Moreover, Lee has shown that repeated application of simple fertilizers was more cost-effective than oleophilic fertilizers. However, in some locations, any additional loss of fertilizer may have negative effects such as stimulation of toxic red tides.

Differences between treatability evaluations will also reflect the degree of biodegradation of the oil prior to the study. The residuals of bioremediation are less amenable to treatment, have a low K_{ow} and their mobility is limited (Angehrn et al. 1998). It has been argued that when the oil has degraded to acceptable biologically benign concentrations, treatment is no longer needed (Lee and De Mora 1999). A chemically based endpoint is more difficult to define than a biologically based endpoint.

Prior to full-scale sediment it is necessary to conduct treatability evaluations. There are advantages to working in a good laboratory but few laboratories are equipped for proper sediment handling. Moreover, it is impossible to complete reproduce certain field conditions such as redox in the laboratory. This paper reviews a few methods of in situ sediment treatments. It is important to optimize the treatment process and to discover any unexpected problems prior to full-scale mobilization.

Methods

Three types of pilot-scale in situ treatments were used.

- 1) One m. square steel frames were used to guide sediment treatment and sampling by divers.
- 2)A box-core incubation allows for conditions as close to ambient as possible but still allows for replication and good controls.
- 3) Full-scale equipment evaluations allows for optimization of the treatment process and clarification of uncertainties not visible in laboratory treatments. Replication and controls are less rigorous than those with the box-core method.

Steel-frame Treatments

On September 2, 1993, four square steel frames were anchored by divers on the sediment surface in the St. Marys River which drains Lake Superior into Lake Huron. This approach was intended to guide future sampling to a small area and hopefully reduce variation associated with sampling. Samples were collected near the frames as controls. The frames were injected with one meter long wands by divers using peristaltic pumps. The injected solution contained 565 g/L of calcium nitrate and a 1% proprietary organic nutrient. On July 18-22, 1994 and again on July 11, 1995 the frames were retreated with calcium nitrate. On October 11, 1995 sediment cores of about 20 cm were collected by divers, extruded into 1 cm sections and analyzed for nitrate, sulphate, TPHs and PAHs.

Box core incubations

On July 11, 1995 six box core liners were placed by divers into sediments in Sault Ste. Marie. The liners were placed to leave a 5 cm lip above the sediments. All sediments in the liners were removed with an air-lift pump. Unlike with the steel frames used earlier, these liners have a bottom. On July 12, 1995 120 liters of sediments were collected mixed, are ated as follows; two controls, two enriched with 3 g NO₃-N/L plus 1% proprietary organic nutrient, two enriched with 6 g NO₃-N/L. The box corers were retrieved Oct. 26, 1995 (108 days) for analysis of PAHs. In this experiment the sediment was isolated but the overlying water was not. Total volume of the box core is 25L (25 cm x 25 cm x 45 cm).

Full-equipment evaluations

For the first large pilot-scale sediment treatments, the vessel the Gander was used to support and transport the sediment injection equipment. This boat was powered by twin Volvo four cylinder diesel motors with two propellers. The boat was 8.2 m long, 3.0 m wide, had a draft of 0.4 m and could be loaded with about 6 tonnes of chemicals and gear. The treatment chemicals were stored in a large tank for pumping into an 8 m wide injection boom. The initial injections were guided by various methods. Cameras observed the depth of the injection boom in the sediments. At first we also had a diver sit on the injection boom and guide the vessel captain by phone. A nitrate electrode was placed on the boom to detect any significant leakage or spill. Sediment cores were also

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collected immediately after sediment treatment for extrusion of 1 cm sections for nitrate analysis by an ion chromatograph.

Total Petroleum Hydrocarbons

A 20 gram sample of wet sediment was extracted with 80 ml dichloromethane for 5 hours on a shaker table. The extract was first dried by filtering it through sodium sulphate and then concentrated by evaporation through a Snyder column. A GC was used to analyze the concentrate.

Results

Steel Frames

The injection of the solution of treatment reagents resulted in considerable release of gases from the sediments. This gas release may have carried some of the treatment chemicals into the water column. The final produced more compacted sediments and a lowering of the sediment surface (> 10 cm). The variability in the injection of calcium nitrate was huge. The distribution varied greatly (not shown). The mean nitrate content of six cores was 111 mg/L with a standard deviation of 119 mg/L. The water clarity at this site was good and the depth of water (4 m) made diving relatively easing. It is hard to imagine a more ideal site for this approach but the initial results were not promising. Attempts to do treatability experiments with small frames inserted into sediments were not satisfactory. There was biodegradation but variability in the responses obscured attempts to optimize the treatment protocol (Table 1, Figure 1).

Table 1 TPH concentrations in in-situ frames (triplicates, mg/g)

Sample	Control	N1	N2	N3	N4	NY1	NY2
Oct. 26, 1993		6.81	4.90	ND	ND	5.51	5.55
Mean July 94	2.98	8.40	6.54	ND	ND	4.09	4.64
SD July 94	0.04	0.29	2.77			0.23	0.28
Mean Oct. 26, 94	4.19	6.21	3.4	3.09	4.41	3.22	2.65
SD Oct 26, 94	1.05	0.8	.31	0.77	1.12	0.37	0.28
Mean July 11, 95	3.35	3.2	1.28	1.74	2.88	0.91	3.28
SD July 11, 95	0.78	1.68	0.43	0.07	1.43	0.28	0.96

SD = standard deviation

In the laboratory with well-mixed samples under more ideal conditions 60% of the TPHs and PAHs had biodegraded in 44 days (TPH data not shown, Figure 2). The much faster biodegradation in the lab probably represents improved mixing of the oxidant and nutrients. We used a radiolabelled phenanthrene to demonstrate that at least for part of the year, at this site cold temperatures do not inhibit biodegradation (Figure 3).

Box Core Liner Incubations

After the less than satisfactory results with the in situ frame we chose to evaluate box core liners as in situ incubation vessels. The mixing on shore adds considerable effort but it overcame the variability problem. It was too late in the project to fully evaluate their utility to optimize bioremediation. There was significant biodegradation of PAHs in three months with the mean and standard deviations from start and end of 69.3 (\pm 2) μ g/g and 56.3 (\pm 1.99) μ g/g.

Full-scale Engineering Pilot-scale Evaluations

Divers and cameras provided the initial guidance to optimize the boat speed, angle of injection, and weight of the boom for optimal penetration of the injection boom. Sediment resuspension was minimal as long as the bottom was flat and the boat did not turn a sharp corner with the boom in the sediments. Initially, the injected sediments acted as a viscous plume maintaining its shape and settling quickly to the bottom. Sediment core analysis added the required detail to optimize the boat speed and chemical pumping rate. A good injection resulted in a subsurface peak of nitrate (Figure 4) that slowly settled deeper into the sediments.

Discussion

The limited biodegradation observed in the small frames in the Sault Ste. Marie site may reflect a combination of high variability of sediments, treatment efficiency including loss of chemicals to surface water, poor mixing of added chemicals and cooler temperatures than the lab. The experiment with the radiolabelled phenanthrene indicated that temperature was not the major limiting variable for bioremediation. Since the frames had open bottoms, the dense chemicals may also have sunk beyond the sampling zone. The frames could not produce significant bioremediation results to optimize treatments within one season. Perhaps with more time the small frame would have produced useful data but most funding agencies require a report within one year.

The box core liners were later used effectively in Japan to evaluate inactivation of phosphorus in sediments (Murphy et al. 1999). For these reactions, redox and pH are critical and in situ incubations are optimal. The most effective aspect of using the box core is improved mixing of the treatment chemicals when the sample is brought to the surface.

The initial developments of the sediment injection equipment were done at Sault Ste. Marie and Hamilton Harbour (Murphy et al. 1995, Murphy et al. 1996). The sites were both awkward in different ways. In Sault Ste. Marie, the deposit of coal tar and wood fibre was as deep as 2 m. It is difficult to bioremediate the surface of these sediments without consideration of the deeper deposit. In Hamilton Harbour, shipping interfered with the sediment treatment and external contaminant sources were not adequately controlled (Irvine et al. 1999). Ideally these issues would have resolved prior to treatment but at least they were discovered prior to full-scale implementation. The full-scale injection equipment has been used effectively to bioremediate coal tar in a beach in Massachusetts (Babin et al. 2001) and to control sulphide odours in Hong Kong (Babin and Murphy 1997, Babin et al. 1999). The third project sediment treatment project with this equipment in Hong Kong is starting in November 2003.

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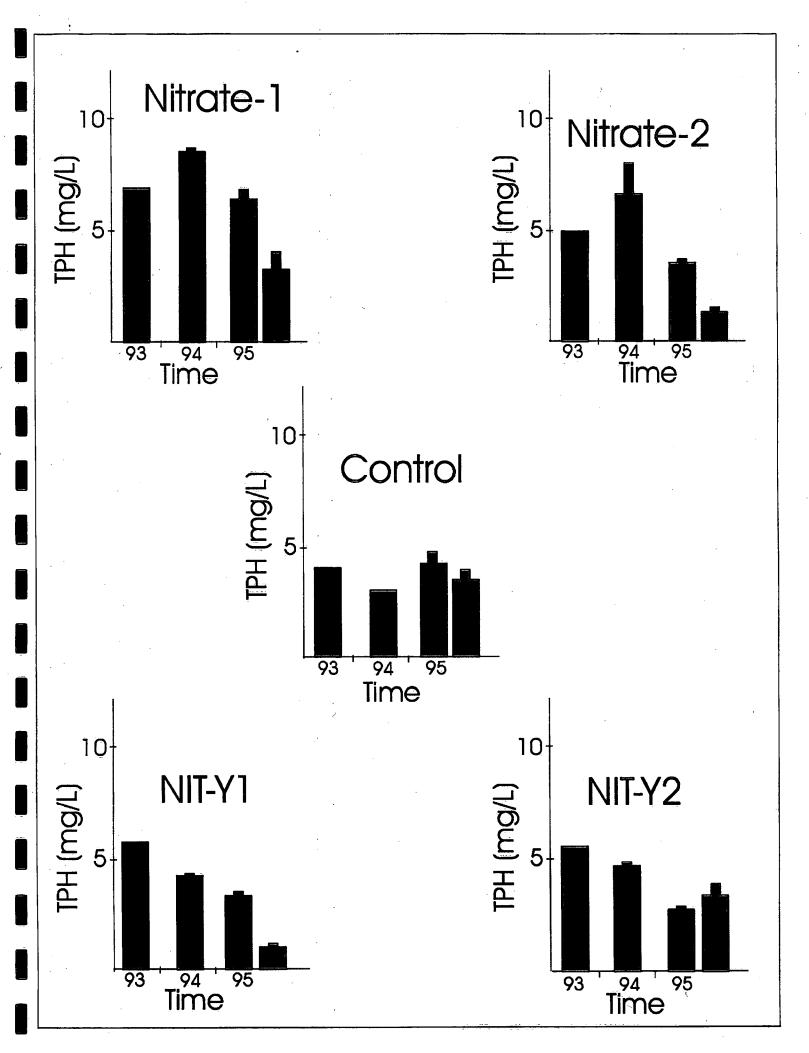
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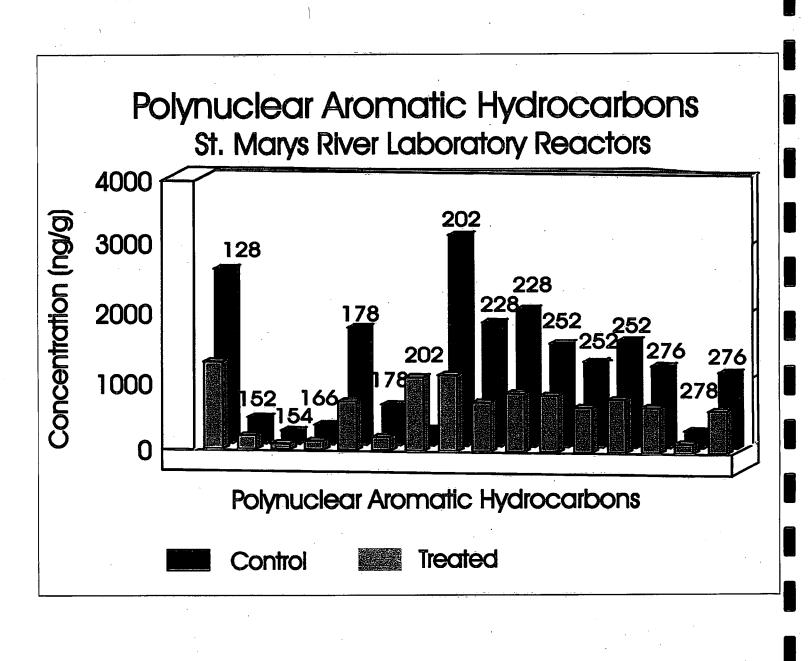
Figure 1 TPH biodegradation in steel frames; Nitrate-1 and Nitrate-2 were injected with calcium nitrate, Nit-Y1 and Nit-Y2 were injected with calcium nitrate and a proprietary organic nutrient.

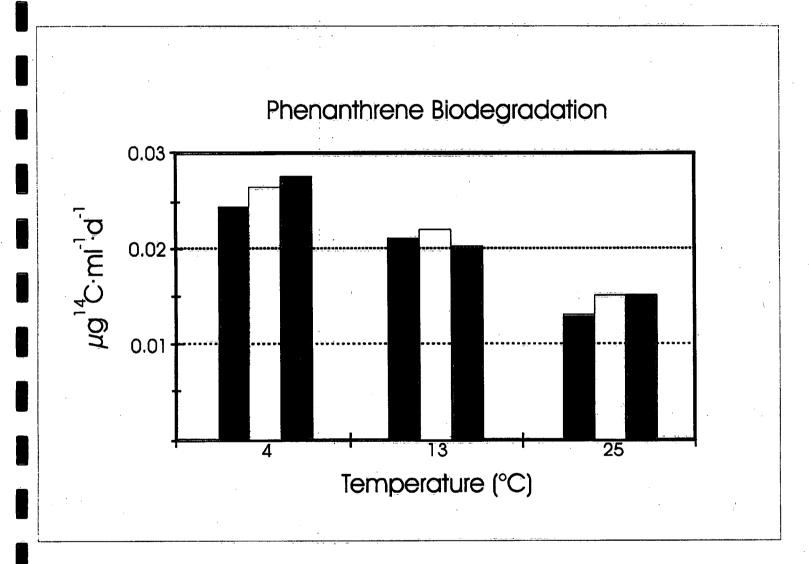
Figure 2 PAH biodegradation in sediments in the laboratory.

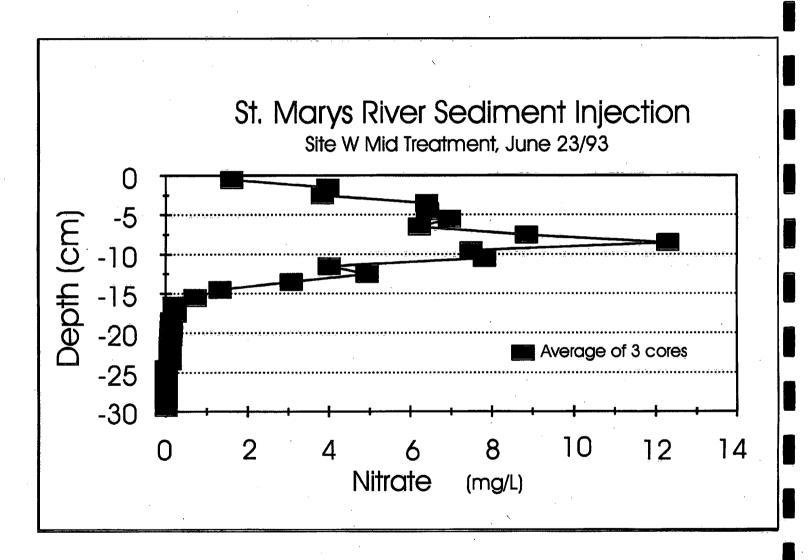
Figure 3 Effect of temperature on bioremediation of phenanthrene.

Figure 4 Injection of calcium nitrate at Sault Ste. Marie.











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