

## Proposal of a sequential treatment methodology for the safe reuse of oil sludge-contaminated soil

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### Abstract

In this study sequential steps were used to treat and immobilize oil constituents of an oil sludge-contaminated soil. Initially, the contaminated soil was oxidized by a Fenton type reaction (13 wt% for H<sub>2</sub>O<sub>2</sub>; 10 mM for Fe<sup>2+</sup>). The oxidative treatment period of 80 h was carried out under three different pH conditions: 20 h at pH 6.5, 20 h at pH 4.5, and 40 h at pH 3.0. The oxidized contaminated sample (3 kg) was stabilized and solidified for 2 h with clay (1 kg) and lime (2 kg). Finally, this mixture was solidified by sand (2 kg) and Portland cement (4 kg). In order to evaluate the efficiency of different processes to treat and immobilize oil contaminants of the oil sludge-contaminated soil, leachability and solubility tests were performed and extracts were analyzed according to the current Brazilian waste regulations. Results showed that the Fenton oxidative process was partially efficient in degrading the oil contaminants in the soil, since residual concentrations were found for the PAH and BTEX compounds. Leachability tests showed that clay–lime stabilization/solidification followed by Portland cement stabilization/solidification was efficient in immobilizing the recalcitrant and hazardous constituents of the contaminated soil. These two steps stabilization/solidification processes are necessary to enhance environmental protection (minimal leachability) and to render final product economically profitable. The treated waste is safe enough to be used on environmental applications, like roadbeds blocks.

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**Keywords:** Soil contamination; Fenton's reaction; Stabilization/solidification; Portland cement; Oil sludge waste

### 1. Introduction

Oil sludge-contaminated soils or sediments are complex mixtures containing many different chemical species with different physico-chemical properties that can show a wide range of toxicity to biota [1]. Among the different chemical compounds present in these hazardous wastes, polycyclic aromatic hydrocarbons (PAHs) were one of the first classes of compounds identified as genotoxics and are often chemicals of concern in environmental waste regulation because of potential adverse effects on public health [2]. This oil contamination of soils may be generated by accidental spills from deliberate dumping, leaching of older landfills or leaking of underground storage tanks. Current clean-up technologies, such as incineration and burial of oil

sludge-contaminated soils in secure landfills, are expensive, and the presence of biorefractory compounds limit bioremediation efficiency. Incorporation of the oil sludge-contaminated soils in a concrete mixture with both economic and environmental benefits could provide a very interesting alternative treatment/fate for these hazardous solid wastes [3]. Thus, in order to minimize potential environmental impact of this waste, it must undergo an efficient chemical treatment, which may be followed by stabilization/solidification technologies to enhance non-leachability properties of the treated waste.

Among the different chemical technologies for treatment of oil sludge-contaminated soil, advanced oxidation (e.g., Fenton's reagent) followed by stabilization/solidification processes with lime–clay and subsequently with Portland cement–lime can lead to the best yields in pollutant degradation/immobilization. In addition, the stabilization/solidification process is cheap compared with many other technologies for treat/dispose solid waste [3].

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In Fenton's process a catalyzed decomposition reaction occurs in which the hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) is decomposed by iron (II) to form hydroxyl radicals ( $\cdot\text{OH}$ ). It is a low cost oxidative process and used in a number of environmental applications, including pre-treatment of industrial wastewaters [4,5], the treatment of water containing dilute concentrations of xenobiotics [6], and the remediation of soils and groundwater [7–9].

Stabilization/solidification processes reduce the mobility of hazardous substances and contaminants in the environment through both physical and chemical means and can be applied both ex situ and in situ. Currently, this process is being used to treat a wide variety of sites containing different classes of contaminants such as polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), organic pesticides and herbicides, halogenated and non-halogenated semivolatile organic compounds and metals [10].

Currently in Brazil, there are no national specific regulations concerning the management of contaminated soils, and they must be considered as solid wastes. According to Brazilian waste regulations [11], solid wastes are classified into two categories: (i) dangerous wastes, and (ii) non-dangerous wastes. Generally, heavy oily wastes derived from petroleum are classified as dangerous wastes. When this type of waste is aggregated with solids, the classification of the solidified waste product is evaluated by experimental protocols of leachability [12] and/or solubility [13]. There are some discussions about environmental concerns regarding the applications of these solidified waste products, but Brazilian National Solid Waste Policy encourages the realization of sound studies for reuse and/or recycling of solid wastes.

In this sense, the objective of this study was to evaluate the effectiveness of the chemical treatment of an oil sludge-contaminated soil by a modified Fenton's reagent followed by two sequential stabilization/solidification processes initially using clay–lime followed by Portland cement–lime in the immobilization of hazardous oil constituents. To achieve this objective, analyses were performed in the contaminated soil, in the Fenton oxidized product, and in a concrete block by means of leachability and solubility tests.

## 2. Materials and methods

### 2.1. Sludge-contaminated soil

The soil was sampled from an oil sludge-contaminated site and prepared for the tests according to the Brazilian standard NBR 6457/86 [14]. Granulometric analysis was made by a sedimentation method after sand siever discard (2.0–0.053 mm) [15]. Contaminated soil physico-chemical characteristics were: pH (4.9); organic matter (13.3%); apparent density ( $\text{g cm}^{-3}$ ) (1.098); clay (30.8%); sand (34.8%); silt (34.4%). The contaminated soil was classified as a dystrophic humic cambisol [16].

### 2.2. Chemical treatment

The previous improved concentrations of the Fenton's reagents used in this step were: 13 wt% for  $\text{H}_2\text{O}_2$  and 10 mM

for  $\text{Fe}^{2+}$ . It is interesting to note that iron soil content was not determined. The treatment period of 80 h was carried out under three different pH conditions: 20 h at pH 6.5, 20 h at pH 4.5, and 40 h at pH 3.0. Adjustment of pH was accomplished by the addition of 5.0 M sulphuric acid or 5.0 M sodium hydroxide. A homogenized mass of 3 kg of oil sludge-contaminated soil from a contaminated site was weighed into a 20 L mixer where 6 L of distilled water was added. After pH adjustment and  $\text{Fe}^{2+}$  addition, the  $\text{H}_2\text{O}_2$  solution was slowly dropped to the contaminated soil. During the addition of the reagents ( $\text{H}_2\text{O}_2$ ), the vial contents were mixed with a mechanical stirrer to ensure homogenization of the mixture. The reaction was stopped after 80 h of oxidation.

### 2.3. Stabilization/solidification treatment

#### 2.3.1. Step 1

The oxidized mixture (9 kg) was placed in a 20 L mixer. Clay (1 kg) and lime (2 kg) were added and stirred for 2 h. The lime was added after 30 min of clay addition. After homogenization, the mixture was allowed to stabilize/solidify for 7 d (exothermic phase).

#### 2.3.2. Step 2

The stabilized solid waste product obtained in Step 1 was re-solidified by mixing with Portland cement (4 kg), sand (2 kg), and water (6 L). After homogenization, the concrete hexagonal block manufactured was allowed to stabilize/solidify for 48 d (curing time) under  $25 \pm 2^\circ\text{C}$  and relative humidity of  $83 \pm 3\%$ .

### 2.4. Leachability and solubility tests

One concrete hexagonal block was fragmented (sieved at 0.1 mm), homogenized and used to carry out leachability and solubility tests. The leachability and solubility tests were carried out according to the Brazilian methodology [12,13]. In the leachability test, a solid sample size of 20 g was placed in a 500 mL bottle, 320 mL of distilled water was added along with a sufficient quantity of acetic acid (0.5N) to adjust the pH to 5.0. The suspension was stirred for 24 h. After filtration with GF membrane (20  $\mu\text{m}$ ), leachable compounds were extracted for analysis. In the solubility test, a solid sample size of 25 g was placed in a 500 mL bottle with addition of 100 mL of distilled water. After 1 h of homogenization, the suspension was allowed to stand for 7 d. After filtration, soluble compounds were extracted for analysis.

### 2.5. Chemical analyses

The samples were fortified with deuterated internal PAH standards and extracted with three aliquots of 20-mL of ultra-pure dichloromethane according to the EPA 3510C protocol [17]. For solid samples a 16-h Soxhlet extraction with dichloromethane was carried out. A Kuderna-Danish concentrator reduced the sample volume to 1-mL. Organic compounds were analyzed on a Hewlett-Packard 5973 GC/MS equipped with a DB-5 column (30 m  $\times$  0.25 mm i.d., 0.25  $\mu\text{m}$  film thickness) in the case of semi-volatile organics and equipped with DB-624 in the case

Table 1  
Analytical results for oil sludge-contaminated soil and soil treated by Fenton's reaction

Parameter (mg kg <sup>-1</sup> )	Oil sludge-contaminated soil	Treated soil (Fenton reaction)
PAH total <sup>a</sup>	85475	5418
Phenols total <sup>b</sup>	344	ND <sup>c</sup> (0.0003)
BTEX total <sup>d</sup>	11198	682
Surfactants	20	ND <sup>c</sup> (0.0005)

<sup>a</sup> PAH total = naphthalene, 1-methyl-naphthalene, 2-methyl-naphthalene, C2-naphthalenes, C3-naphthalenes, C4-naphthalenes, acenaphthylene, acenaphthene, fluorene, C1-fluorenes, C2-fluorenes, biphenyl, phenanthrene, C1-phenanthrenes, C2-phenanthrenes, C3-phenanthrenes, anthracene, fluoranthene, dibenzothiophene, C1-dibenzothiophenes, C2-dibenzothiophenes, pyrene, C1-pyrenes, C2-pyrenes, benzo(a)anthracene, crysene, C1-crysenes, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, perylene, indeno(1,2,3-CD)pyrene, dibenzo(a,h)anthracene, benzo(g,h,i)perylene.

<sup>b</sup> Phenols total = phenol, 2-methylphenol, 3-methylphenol, 4-methylphenol, 2-chlorophenol, 2,6-dimethylphenol, 2-ethylphenol, 2,5-dimethylphenol, 2,4-dimethylphenol, 4-ethylphenol, 3,5-dimethylphenol, 2,3-dimethylphenol, 3,4-dimethylphenol, 2-isopropylphenol, 2,3,5-trimethylphenol, 4-chloro-3-methylphenol, 2,6-dichlorophenol, 2,4-dichlorophenol, 2-nitrophenol, 2,4,6-trichlorophenol, 4-nitrophenol, 2,4,5-trichlorophenol, 2,3,4,6-tetrachlorophenol, pentachlorophenol.

<sup>c</sup> ND, not detected above concentration showed in parenthesis (mg kg<sup>-1</sup>).

<sup>d</sup> BTEX total = benzene, toluene, ethylbenzene, *m,p*-xylenes, *o*-xylene.

of volatile organics according to the EPA 8270C and 8260B protocols [17]. The injector temperature was 280 °C (splitless). Chromatographic conditions included an initial oven temperature of 40 °C (37 °C for volatiles), a 1-min isotherm, a program rate of 6 °C/min, and a final oven temperature of 280 °C (200 °C for volatiles) with an isotherm of 8 min. The mass spectrometer operated in ion selective mode (less than 3 cycles/s). Other analyzed parameters in solids were performed according to the procedure established by USEPA SW 846 Methods [17]. For others parameters analyzed in aqueous solution, standard procedures were adopted [18].

### 3. Results and discussion

#### 3.1. Chemical oxidation

Table 1 shows quantified organic chemicals in the contaminated soil before and after the chemical oxidation.

Table 2  
Analytical results for concrete leaching and solubility tests with their respective limit values according to the Brazilian standards

Parameter (mg L <sup>-1</sup> )	Leachability test	ABNT 10004 Leaching limit value	Solubility test	ABNT 10004 Solubility limit value
PAH total <sup>a</sup>	ND <sup>b</sup> (0.00001)	NL <sup>c</sup>	ND <sup>b</sup> (0.00001)	NL <sup>c</sup>
BTEX total <sup>d</sup>	ND <sup>b</sup> (0.0001)	NL <sup>c</sup>	ND <sup>b</sup> (0.0001)	NL <sup>c</sup>
Hardness <sup>e</sup>	NL <sup>c</sup>	NL <sup>c</sup>	1983	500

<sup>a</sup> PAH total = naphthalene, 1-methyl-naphthalene, 2-methyl-naphthalene, C2-naphthalenes, C3-naphthalenes, C4-naphthalenes, acenaphthylene, acenaphthene, fluorene, C1-fluorenes, C2-fluorenes, biphenyl, phenanthrene, C1-phenanthrenes, C2-phenanthrenes, C3-phenanthrenes, anthracene, fluoranthene, dibenzothiophene, C1-dibenzothiophenes, C2-dibenzothiophenes, pyrene, C1-pyrenes, C2-pyrenes, benzo(a)anthracene, crysene, C1-crysenes, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, perylene, indeno(1,2,3-CD)pyrene, dibenzo(a,h)anthracene, benzo(g,h,i)perylene.

<sup>b</sup> ND, not detected above concentration showed in parenthesis (mg L<sup>-1</sup>).

<sup>c</sup> NL, not listed in ABNT protocol.

<sup>d</sup> BTEX total = benzene, toluene, ethylbenzene, *m,p*-xylenes, *o*-xylene.

<sup>e</sup> mg L<sup>-1</sup> of CaCO<sub>3</sub>.

Due to the high soil-organic contents, oxidation prior to stabilization/solidification processes must be realized because greases, phenols and oils have a significant detrimental effect on the physical properties of solidified/stabilized wastes [19]. Preliminary oxidation study showed that the high reagent concentrations tested, i.e., H<sub>2</sub>O<sub>2</sub> (13%) and Fe<sup>2+</sup> (10 mM), were more efficient in the oxidation process, which is in conformity with others studies [20,21]. Probably, iron soil content has influenced Fenton oxidation kinetics, as well as soil organic contents. For this reason, each specific soil must be tested to find effective ratios between Fenton's reagents in view to improve the contaminant degradation. In the tested experimental condition, phenolic compounds and surfactants were undetected after oxidation treatment, while low concentrations were found for the PAH and BTEX compounds.

The higher degradation rate of contaminants under a low pH regime may be due to the highly reducing conditions. Thus, it was reported that low pH conditions favored the oxidation of organic compounds, and hypothesized that Fe(OH)<sup>+</sup>, formed at a pH range of 2–4 units, has a higher activity than Fe<sup>2+</sup> in Fenton's reaction and leads to the improved compound degradation in this pH range (including aliphatics hydrocarbons) [20,22,23]. On the other hand, oxidation of the aromatic compounds required less iron and less H<sub>2</sub>O<sub>2</sub> than oxidation of the aliphatic compounds, while proceeding more effectively at near-neutral pH [23]. For these reasons, a pH gradient was used in our study (from 6.5 to 3.0).

Concerning PAH residues after oxidation, phenanthrenic compounds appear to be the most resistant family, while other PAH families were almost completely degraded. PAH residues in treated soil may have regulatory significance and implications for remediation techniques, cleanup standards, and risk assessments. In this sense, it is recognized that hydrophobic organic compounds are associated primarily with soil natural organic matter [24], which may limit oxidation efficiency. Thus, remediation efficiency is limited by the contaminant availability, which in turn is dependent on contaminant chemical properties, soil characteristics, and soil-contaminant age [25].

#### 3.2. Stabilization/solidification processes

Table 2 shows analytical results with their respective limit values according to Brazilian standards for concrete leachability

and solubility tests carried out with samples oxidized with  $\text{H}_2\text{O}_2$  (13%) and  $\text{Fe}^{2+}$  (10 mM).

Previous studies showed that organic pollutants are not immobilized by solidification process using lime or Portland cement as only solidificant agent [26–28]. Thus, in the present proposal the first step encompasses pollutant physico-chemical adsorption by clay–lime. Many studies showed the organic pollutants' behavior when in contact with clay particles, where chemical reactions occur and promote solidification process with cement [28–31].

Comparison of results from Tables 1 (data after oxidation) and 2 show that PAHs and BTEX compounds decrease after stabilization/solidification (S/S) processes. This can be explained by the dilution which occurs in this step (where clay and lime were added) and by immobilization promoted by the lime and cement. In this sense, lime has been used alone for S/S processes applied to high oil or tar content wastes [32].

Despite stabilization/solidification processes that enabled the classification of the initial waste to be changed from dangerous (Class I) to non-dangerous (Class II), the mass/volume ratio of residual product increased after treatment (3 kg of waste yields 20 kg of commercial concrete blocks). This fact could be a economic problem when the stabilized/solidified product is landfilled, but if we consider reuse of this product as concrete roadbed blocks, the final economic balance is positive. For example, costs of oil sludge-contaminated soil landfilling are usually in the order of US\$ 100–200 per tonne of waste, while 1 tonne of concrete roadbed blocks (with 15% of waste constitution) yields about US\$ 300. Regarding the ecotoxicity potential of samples, the initial waste showed a high PAH level, as well high concentrations of phenolic compounds, whereas the final product classification is due to its Hardness, recognized as less toxic. Hardness is caused by a variety of dissolved polyvalent metallic ions, predominantly calcium and magnesium cations, although other cations, e.g., barium, iron, manganese, strontium and zinc, also contribute. These ions are natural constituents of the soil, lime, and cement. There does not appear to be any convincing evidence that water hardness causes adverse health effects in humans [33]. Potential of oil leaching components in the long-term must be evaluated by a temporal series of lixiviation tests to ensure environmental protection.

#### 4. Conclusions

The experimental results show that the sequential treatment/immobilization processes used in this study can represent an interesting alternative for the final disposal of oil sludge-contaminated soil. The mixing of oxidation treated soil with clay, lime, sand and Portland cement to manufacture concrete roadbed blocks allowed the classification of the initial waste to be changed from dangerous to non-dangerous, with immobilization of all toxic organic constituents, as observed after leachability and solubility test procedures carried out according to Brazilian waste regulations. The two steps of stabilization/solidification ensure low lixiviation potential, as well as economic return, because concrete roadbed blocks are commercial products. The major problem related to these product applications is the poten-

tial of oil leaching components in the long-term and the consequent environmental impacts in terms of public health and/or ecotoxicological perturbations of terrestrial or aquatic ecosystems. Thus, further efforts should be made to verify whether the sequential chemical treatment proposed ensure long-term waste immobilization and whether this process can be applied to other organic solid wastes.

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