



Application of a three-stage remediation process to cleanup petroleum-hydrocarbon contaminated sediments

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ABSTRACT

Sediments are usually the sinks for many waterborne pollutants, and thus, contaminated sediments need to be remediated to prevent the release of pollutants into the water bodies. The objective of this study was to develop a three-stage system to cleanup total petroleum hydrocarbons (TPH)-contaminated sediments. Sediments were collected from the Kaohsiung harbor with a TPH concentration of 8,105 mg/kg. Results from the sediment analyses show that sediments were mainly fine-grained silts, and the sediment organic matter content was about 19%. Thus, the surfactant washing method became an optimal option for the cleanup of TPH-contaminated sediments at this site. The three-stage scheme included the river water (RW) washing stage followed by the surfactant (Triton X-100) washing and Fenton-like oxidation stages. Batch experiments were conducted to evaluate the TPH removal efficiency by RW washing and surfactants washing with initial Triton X-100 concentrations of 0.5, 1, and 5% (w/w). In these oxidation experiments, three different hydrogen peroxide (H₂O₂) concentrations (0.6, 1.05, and 6 vol.%) were used to evaluate the TPH oxidation efficiency. Based on the results of the batch experiments, the three-stage operational process was performed using the optimal conditions from the batch experiments. The three-stage process was performed using 10 pore volumes (PVs) of RW washing followed by 30 PVs of Triton X-100 (0.5%) washing and 20 PVs of hydrogen peroxide (6%) oxidation. Results show that approximately 6% of TPH was removed after the RW washing stage. Up to 71% of TPH was removed by surfactant washing through mobilization and solubilization mechanisms. Approximately, 8% of TPH was removed through the Fenton-like oxidation, and a total of 86% of TPH was removed after the three-stage processes. This implies that the Fenton-like oxidation could be used as a polishment method for the TPH-contaminated sediment treatment. Results indicate that the proposed sediment treatment scheme could provide an efficient and cost-effective alternative for TPH-contaminated sediment remediation.

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Keywords: Fenton-like oxidation; River water washing; Sediment; Surfactant washing; Total petroleum hydrocarbon

1. Introduction

Sediments contaminated by organic chemicals are a serious environmental problem worldwide. The US Environmental Protection Agency investigated more than 19,000 sediment sampling stations across the country and found that about 73% of them fell within Tier 1 and Tier 2 sites, which indicated that adverse effects on ecosystem and human health might occur at these sites [1]. Kaohsiung Harbor is the largest international harbor in Taiwan and receives water flows from four contaminated rivers, which includes Love River, Canon River, Jen-Gen River, and Salt River. The major pollution sources of these four rivers included domestic, agricultural, and industrial wastewaters from the upstream catchments [2–5]. Thus, Kaohsiung Harbor receives pollutants from the four rivers continuously. Results from our previous investigation indicate that the sediments of Kaohsiung Harbor at the river outfalls are contaminated by petroleum hydrocarbons, probably due the illegal discharges of engine or lubricant oils from the surrounding machine shops or ship yards [6]. Sediments are the sinks for organic pollutants and heavy metals in water bodies, and the accumulated pollutants on sediments would cause the deterioration of the sediment quality. Furthermore, the releases of pollutants from the contaminated sediments would also result in significant adverse impacts on the water quality. Thus, the contaminated sediments at the Kaohsiung Harbor need to be cleaned up to protect the surrounding ecosystem.

Different remedial approaches have been proposed to treat organic-pollutant contaminated sediments [7]. Among them, sediment washing and chemical oxidation have been considered as feasible remedial technologies. Furthermore, the treatment train concept is becoming increasingly popular alternative for the treatment of organic contaminated media. Two or more innovative and established technologies may be used together in treatment trains, which are either integrated processes or a series of treatments that are combined in sequence to provide the necessary treatment. Some treatment trains are employed when no single technology is capable of treating all the contaminants in a particular medium. In addition, a treatment train might be used to render a medium more easily treatable by a subsequent technology, reduce the amount of waste that requires further treatment by a

subsequent and more expensive technology, or minimize the overall cost of the treatment [8].

Surfactants are surface-active agents and amphiphilic compounds with hydrophilic and hydrophobic portions [9,10]. Literatures indicated that surfactant washing can be effective for hydrophobic materials (e.g. petroleum hydrocarbons) [11,12]. Sediment washing is based on the desorption of pollutants from contaminated sediments through the action of simple water or organic surfactants, which may be non-ionic or anionic. Surfactants are interesting remediation agents because they have unique characteristics that change the surface property of liquids. Above a specific concentration, the critical micelle concentration (CMC), the surfactant molecules aggregate to form micelles with a hydrophobic head occupying the core and a hydrophilic tail directed at the exterior surface. Since micelles provide an organic microsphere in an aqueous solution, contaminants such as petroleum hydrocarbons can be easily solubilized within their micellar phase [13]. The washing treatment is cost effective and relatively fast compared with other remediation technologies, thereby having potential to be applied in treating and removing a large amount of pollutant [14].

Chemical oxidation with hydrogen peroxide (H_2O_2) can be direct and/or involve generation of free radicals. Hydrogen peroxide is a powerful oxidizing agent, which can be delivered deep into the ground by using soil mixing techniques or injected groundwater amendments [15,16]. Recently, there are many studies published on the Fenton-like remediation of petroleum-contaminated sediment [17–19]. Fenton-like processes occur by utilizing naturally occurring mineral irons, such as goethite and hematite, and in soil have focused on the process chemistry that most effectively destroys and mineralizes biorefractory contaminants [15,20]. In addition, Fenton-like reactions have been investigated specifically for the treatment of petroleum fractions and related compounds in soils [21]. The application of Fenton-like chemical oxidation process for the remediation of contaminated soils has gained more attention in recent years. Several studies observed that various organic contaminants in soil could be oxidized by hydrogen peroxide in the presence of iron oxide mineral. This has been identified as a potentially effective technology in the treatment of

chlorinated solvents and petroleum-hydrocarbon pollutants.

In this study, three-stage remediation process (river washing, surfactant washing, and chemical oxidation) was used to remove total petroleum hydrocarbons (TPH) from oil-contaminated sediments collected from a sediment-polluted site in Kaohsiung Harbor. The objectives of this study were to (1) evaluate the efficiency of using river water (RW) and surfactant washing processes on TPH removal from sediments, (2) assess the feasibility of Fenton-like oxidation for TPH-contaminated sediments remediation, and (3) determine the optimal operational conditions of three-stage remediation process to clean up TPH-contaminated sediments. Results from our preliminary investigation indicate that the sediments collected from the Salt River outfall contained higher concentrations of TPH. Thus, sediments collected from the Salt River outfall were used for the following treatment study.

2. Materials and methods

2.1. TPH-contaminated sediment

In this study, the sediment samples were collected from the outfall of the Salt River and Canon River in the Kaohsiung Harbor. Fig. 1 presents the sampling

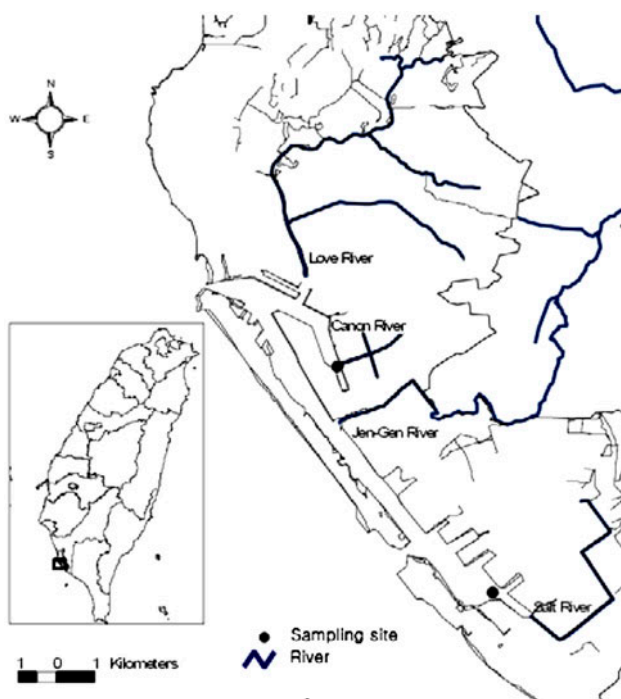


Fig. 1. Site map showing the Kaohsiung Harbor and sediment sampling site.

sites in the Kaohsiung Harbor. Sediment samples were collected using a bottom sampler. The samples were air-dried, passed through a 2 mm sieve for fully homogenized, and kept refrigerated at 4°C until analyzed [4,15]. The sediment samples were analyzed to determine their characteristics including parameters of pH value, oxidation-reduction potential (ORP), sediment organic matter (OM), and particle size. The sediment pH and ORP value were measured using a mixture of sediment and deionized water (1:1, w/v) with a glass electrode. The OM was evaluated using Walkley–Black titration method. Sediment particle sizes were determined with a particle size counter (Coulter LS 100, Particle Size Analyzer).

2.2. RW and surfactant washing experiments

A biodegradable and non-ionic surfactant (Triton X-100) was used in the batch experiment. Table 1 shows the characterization of Triton X-100 (TX-100). TX-100 was purchased from Sunshine Makers, Inc. (USA), which has a molecular formula as $C_8H_{17}C_6H_4O(CH_2CH_2O)_{9.5}H$, an average molecular weight of 625 g/mol, a CMC of 0.15 g/L, hydrophile-lipophile balance number (HLB) of 13.6, and a cost of 23 USD/kg. Fill-and-draw batch experiments were conducted to evaluate the TPH removal efficiency by RW washing and surfactants washing with an initial surfactant concentrations of 0.5, 1, and 5% (w/w). In this experiment, an enclosed reactor (40 mL glass bottle) contained 5 g of TPH-contaminated sediments and 10 mL (one pore volume [PV]) of TX-100 solution. The reactor was shaken in a shaker at 250 revolutions per min (RPM) for 5 min to simulate sediment washing process. These experiments were conducted at room temperature, and duplicate bottles were sacrificed at 5, 10, 15, 20, 30, 45, and 60 PVs for the analyses of TPH concentrations. TPH concentrations were determined by shake extraction method for 24 h with mixture of n-hexane/acetone (v/v, 1:1) and analyzed by a Hewlett Packard 6890 gas chromatography equipped with a flame ionization detector and a DB-1 capillary column (0.32 mm × 30 m). The injector temperature was 300°C and the detector temperature was 350°C. The oven temperature was programmed to increase from 50°C (5 min) to 350°C (10 min) at 10°C/min [15].

2.3. Fenton-like oxidation experiments

In this Fenton-like oxidation experiments, three different hydrogen peroxide (H_2O_2) concentrations (650 (0.6 vol.%), 1,143 (1.05 vol.%), and 6,500 mM (6 vol.%)) were used. This batch experiment was performed to

Table 1
Characteristics of surfactant Triton X-100

Compound	Molecular formula	MW (g/mol)	CMC (g/L)	HLB	Cost (USD/kg)
Triton X-100	$C_8H_{17}C_6H_4O(CH_2CH_2O)_{9.5}H$	625	0.18	13.4	23

evaluate the effect of H_2O_2 concentrations on the efficiency of Fenton-like reaction. The groups of batch experiments were conducted in 40 mL glass bottles with teflon caps batch containing 5 g of TPH-contaminated sediments (with initial TPH concentration of 8,691 mg/kg) and 15 mL of H_2O_2 solution. The experiments were conducted at room temperature, and duplicate bottles were sacrificed at 20, 40, 60, 120, and 180 min time points for the analyses of TPH concentrations during the reaction. Samples were spun on a Hettich-Zentrifugen EBA 21 Centrifuge for 5 min at 2,500 rpm, and stored until analysis at 4 °C in the dark.

2.4. Three-stage treatment system

This experiment was conducted to evaluate the effectiveness of applying the three-stage treatment train scheme on the remediation of TPH-contaminated sediments. The first and second stages of the treatment train system were the washing processes applying RW and biodegradable surfactant TX-100. The 40 mL glass bottles were used in this study containing 5 g of TPH-contaminated sediments (with initial concentration of 8,691 mg/kg). A fill-and-draw method was simulated using the washing processes by adding different PVs of RW followed by surfactant washing solution. After RW and surfactant solution washing treatment, the third stage of treatment train scheme was the chemical oxidation applying Fenton-like process as the oxidation treatment. The bottles were injected with 6% H_2O_2 and the experiment was conducted at room temperature. Sediment samples were collected at different time points (e.g. 0, 10, 20, and 30 min) for the analyses of TPH concentrations.

3. Results and discussion

3.1. Sediment characterization

In this study, the physical-chemical characteristics of the collected Salt River sediments are presented in

Table 2. Results from the sediment analyses show that sediments were mainly silt loam (14% of sand, 67% of silt, and 18.0% of clay). This might be due to the high precipitation resulted high river flows, which made the silts become the main components of sediments at the river outfall. Results show that the sediment OM content was 19%. Higher OM concentrations were probably due to the discharges of organic pollutants from adjacent sewage discharge pipes or illegal industrial wastewater discharges. Results reveal that chemical oxidation method would not be the appropriate treatment option for the treatment of high OM-contained silts due to the low oxidation efficiency. This makes the surfactant washing method become an optimal option for the cleanup of TPH-contained sediments [11,12].

3.2. RW and surfactant washing experiments

In the batch study, RW and surfactant washing experiments were performed to evaluate the TPH removal efficiency of sediment on TPH-contaminated sediment remediation. Fig. 2 presents the TPH removal efficiencies using RW and TX-100 (0.5, 1, and 5%) as washing reagents for sediment washing. Fluctuations of the removal efficiencies were observed during the fill-and-draw experiments. This might be due to the occurrence of sediment particle aggregation of the fine-textured sediments, which limited adsorbed pollutants from dissolution and removal. Results show that approximately 13.4% of TPH can be removed when 60 PVs of the RW were applied for the washing process. The surfactant washing experiments show that approximately 95, 100, and 100% of TPH can be removed when 60 PVs of surfactant solution with 0.5, 1, and 5% of TX-100 were applied for sediment washing, respectively. Results also show that 100, 93.7, and 72.6% of TPH can be removed when 30 PVs of surfactant solution with 5, 1, and 0.5% of TX-100 were applied for sediment washing, respectively.

Table 2
Characteristics of collected sediments

pH	ORP (mV)	Sediment OM (%)	TPH (mg/kg)	Sand (%)	Silt (%)	Clay (%)	Type
5.9	-265	19	8,105	15.7	68.3	16.0	Silt loam

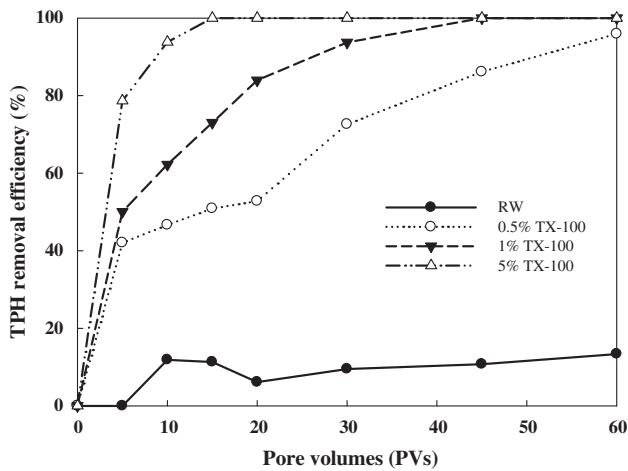


Fig. 2. TPH removal efficiencies using RW, 0.5, 1, and 5% of TX-100 as washing reagents for sediment washing.

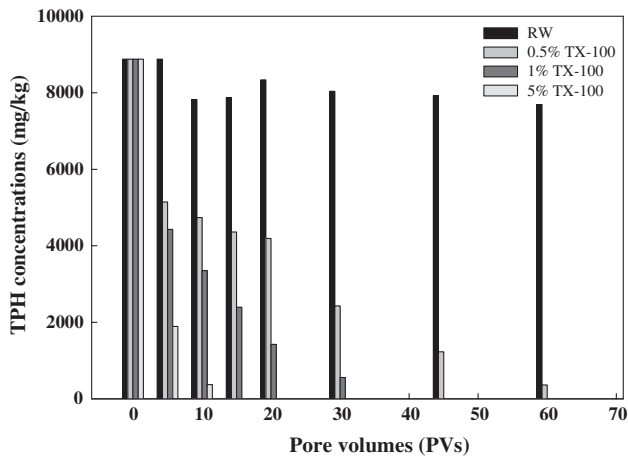


Fig. 3. Variations in remaining TPH concentrations in sediments when different PVs of RW and surfactant solution were applied for sediment washing.

Fig. 3 presents the variations in remaining TPH concentrations in sediments when a different PV of TX-100 solution was applied for sediment washing. Results show that TPH concentration dropped to 279 mg/kg when 60 PVs of 0.5% TX-100 solution were applied for sediment washing. This indicates that low TX-100 concentration (<1%) and low volume of washing solution (<60 PVs) would be able to achieve high TPH removal efficiency. Compared to the results obtained from literature [6], higher percentage of TPH removal was observed in this study. This was due to the fact that CMC value of TX-100 was lower compared with other surfactants applied by other researchers [8]. It indicates that TX-100 could increase the TPH contamination solubility and detergency, and

thus, results in higher removal efficiency [22]. To optimize the performance of sediment washing, results show that the appropriate amounts of surfactant application is critic. Appropriate surfactant injection into the contaminated system would enhance the solubilization and mobilization of the contaminant in the water phase. Application of sediment washing by TX-100 would increase the efficiency of contaminant removal from the highly contaminated sediments. In the field application, the decrease in sediment permeability might occur after surfactant washing process due to the remained surfactant in the sediment pores. RW washing could be applied to replace surfactant for the second phase of washing. Thus, application of RW washing (or extraction) after the TX-100 washing could further improve the efficiency of TPH removal, and also prevent the possible clogging problem due to the usage of surfactant.

3.3. Fenton-like oxidation experiments

In this experiment, degradation of TPH by oxidation process was evaluated. Fig. 4 presents the efficiencies of TPH removal vs. reaction time with three different initial H_2O_2 concentrations (650, 1,143, and 6,500 mM). Fig. 5 shows the variations in remaining TPH concentrations in sediments when different H_2O_2 concentrations were applied for sediment oxidation. Results show that TPH dropped from 8,105 to 5,889 mg/kg after 180 min of oxidation process with 650 mM of H_2O_2 addition and the TPH removal efficiency was approximately 27.3%. When 1,143 mM of H_2O_2 was applied, the TPH concentrations decreased from 8,105 to 5,457 mg/kg (approximately, 32.7% of

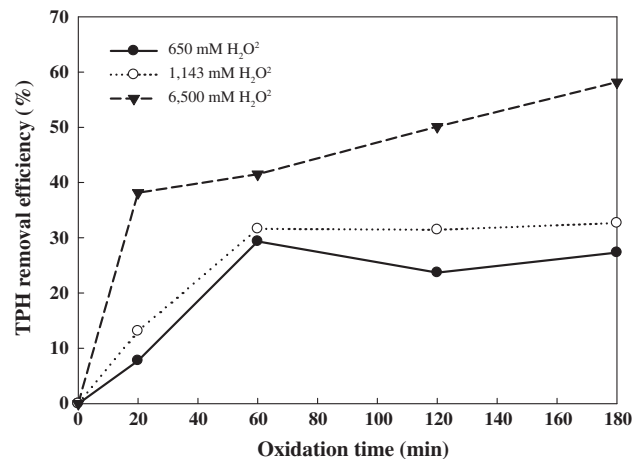


Fig. 4. TPH removal efficiency vs. reaction time with different H_2O_2 concentrations of 650, 1,142, and 6,500 mM.

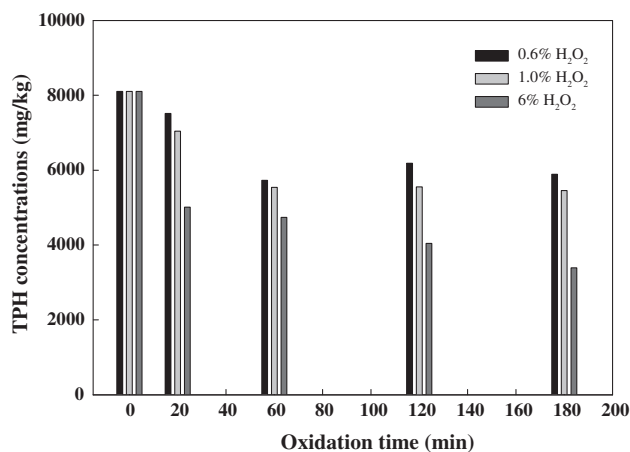


Fig. 5. Variations in remaining TPH concentrations in sediments when different H₂O₂ concentrations were applied for sediment chemical oxidation.

TPH removal) after 180 min of reaction. When 6,500 mM of H₂O₂ was used for the sediment oxidation process, the TPH concentrations decreased from 8,105 to 3,392 mg/kg (approximately, 58.2% of TPH removal) after 180 min of reaction. Results show that TPH removal can be significantly increased with an increase in H₂O₂ concentration with initial TPH concentration of 8,105 mg/kg. This indicates that the TPH oxidation can be enhanced under conditions of higher H₂O₂ concentration. Researchers have reported that high concentrations of H₂O₂ (generally above 294 mM) are required for site application because various OMs in soil would quench some part of supplied H₂O₂ [15,20]. During the oxidation process, an increase in the ORP values was also observed. When 6,500 mM of H₂O₂ was used for the sediment oxidation process, the ORP values increased from -149 to 356 mv after 180 min of reaction. The ORP is a measure of the oxidizing environment and can be used to determine oxidant movement in the field. Positive values of ORP reflect oxidizing conditions and generally coincide with the oxidant movement. Results from the oxidation experiments also show that the supplement of higher H₂O₂ concentrations is a necessity to remediate highly TPH-contaminated sediments. Results also imply that higher H₂O₂ concentrations would cause higher TPH degradation rate and higher TPH removal efficiency under the condition of sufficient reaction time. Thus, the oxidation process could be an effective method to oxidize TPH on site. Results suggest that, for the future practical application, a sequential addition of high concentration of H₂O₂ can be conducted to provide a stronger oxidizing power to obtain the desired treatment efficiency. Moreover, the produced

oxygen from the H₂O₂ oxidation process can act as the electron acceptor source for the enhancement of aerobic biodegradation of TPH in the system.

3.4. Three-stage treatment system

Percentage of TPH removal vs. different PVs of applied RW, surfactant (0.5% of TX-100), and H₂O₂ (6%) is shown in Fig. 6. The TPH removal via the RW washing process was approximately 6% (total TPH removal efficiency of three-stage treatment train system) after 10 PVs of RW washing. After the RW washing process, 0.5% of TX-100 surfactant was added into the reactor for the second-stage sediment remediation process. In this treatment stage, the TPH removal efficiency was approximately 71%. In the third-stage of the treatment train process (Fenton-like oxidation process), approximately 8% of the TPH removal (total TPH removal efficiency of three-stage treatment train system was 86%) was obtained after oxidation, while the TPH concentrations dropped from 8,105 to 1,134 mg/kg at the end of the oxidation process. Table 3 presents the percentage of total TPH removal after the application of the three-stage treatment train system. Results indicate that approximately 78% of TPH could be removed after RW and TX-100 (0.5%) washing (Stages 1 and 2). The major TPH removal process during the first-stage process was desorption mechanism. During the second-stage process, the main TPH removal mechanisms were desorption and solubilization. The Fenton-like (6% of H₂O₂) oxidation process (Stage 3) was able to remove another 8% of the remaining TPH. During the third-stage process, the main TPH removal mechanisms were desorption and oxidation. Thus, complete TPH removal could be obtained using the three-stage treatment scheme. In the future practical design, a RW washing followed by surfactant and RW washing processes can be developed for field application. Thus, the total cost of sediment remediation can be minimized. RW washing can be also applied after the TX-100 washing to prevent the possible clogging problem due to the usage of surfactant. The three-stage RW-surfactant-oxidation treatment train scheme would be expected to provide a more practical and efficient alternative for TPH-contaminated sediment remediation. However, results also indicate that although H₂O₂ can be used as an oxidant alone, the oxidation reaction is not kinetically fast enough to degrade many hazardous organic contaminants with low H₂O₂ concentration. Thus, when H₂O₂ is used for on site application, high concentration of H₂O₂ is required because various OMs in river sediments could also quench H₂O₂. Furthermore,

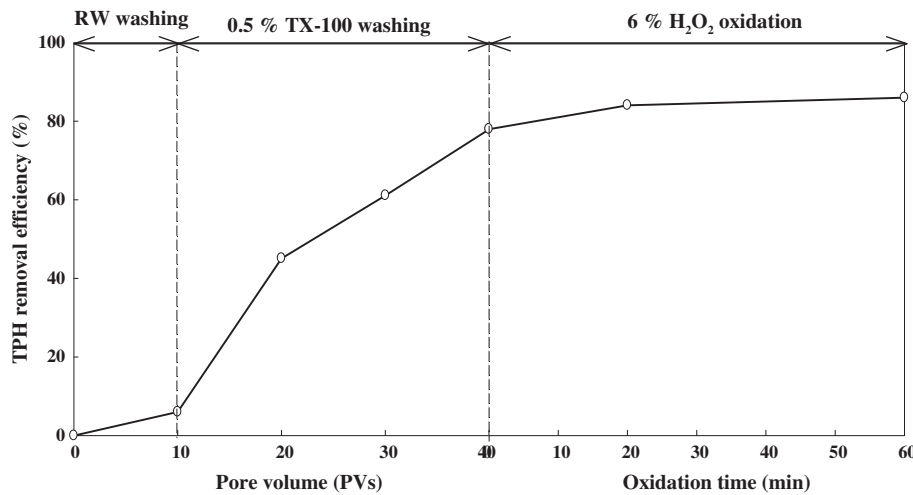


Fig. 6. Percentage of TPH removal vs. different PVs of applied RW, surfactant (0.5% of TX-100), and H₂O₂ (6%).

Table 3

Calculated percentage of total TPH removal after the application of the three-stage treatment train system

Stage	Reaction mechanism	TPH conc. (mg kg ⁻¹)		Total TPH removal efficiency (%)
		Initial	Final	
1	RW washing	8,105	7,615	6
2	Surfactant washing	7,615	1,786	78
3	Fenton-like oxidation	1,786	1,134	86

results from this study also reveal that the presence of H₂O₂ might be a feasible method to enhance the aerobic bioremediation efficiency of petroleum-hydrocarbon contaminated sediments.

4. Conclusions

In this study, a three-stage treatment train scheme has been developed to remediate TPH-contaminated sediments. Conclusions obtained from this study included the following:

- (1) Application of RW washing before the surfactant washing process as stage 1 could remove 6% of sediment TPH. Thus, the amount of surfactant usage can be reduced.
- (2) Up to 71% of TPH removal can be obtained using surfactant washing through mobilization and solubilization. This indicates that surfactant washing using Triton X-100 surfactant is a promising technology to remediate TPH-contaminated sediments.
- (3) Results show that 8% of TPH can be oxidized through the Fenton-like oxidation process.

This indicates that the Fenton-like oxidation can be used as a final polishing stage for the TPH-contaminated sediment cleanup process. Furthermore, the remaining oxygen from the Fenton-like process can act as the oxygen source for the enhancement of aerobic biodegradation of TPH in sediments.

- (4) Up to 86% of TPH removal efficiency could be obtained after the three-stage treatment processes. The proposed three-stage treatment train scheme would be expected to provide a more efficient and cost-effective alternative to remediate TPH-contaminated sediments. The developed remedial system has the potential to be applied to remediate other organic pollutant contaminated sites.

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