

Treatment of Permanganate Oxidation and Bioremediation on Petroleum Hydrocarbons Contaminated Soil and the Effect on Soil Function

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Abstract: The impact of potassium permanganate oxidation-bioremediation on soil function can be divided into three stages: the oxidation stage, the transition stage, and the bioremediation stage. In order to obtain a deeper revelation of the effect on soil function, the nitrogen, phosphorus, organic matter (OM), dissolved organic carbon (DOC) and petroleum hydrocarbons (TPH) during these three phases of remediation have been investigated. Potassium permanganate (PP) was consumed after 24 hours' reaction. During this phase, a larger removal rate of TPH was achieved at a PP molar concentration of 0.05 and 0.1 mol/L and under weak acidic or basic conditions. 18%-61% of TPH was removed in 24 hours. PP has a strong impact on soil functionality. Addition of oxidation agent largely decreased DOC amount in soil. However, DOC and the proportion of active OM in soil increased as the connection time (phase two) was prolonged. DOC amount was 172% increased after 60d. After the three phases' combined remediation, more than 70% of the TPH in soil was reduced while the maximum removal rate was 97.35%. The concentration of the C₁₀-C₁₂ segment has significantly diminished to the point of near disappearance, while the C₁₉-C₄₀ segments have experienced an approximate 40% reduction. The removal rate for high-carbon chain segments remains satisfactory. Addition of tween-80 effectively increased the solubilization and removal rate of TPH while introduced DOC into the reaction system. Moreover, the previous consumption of oxidizers is relatively slow, making it an ideal additive for high organic pollutant-low soil organic matter affinity. Results showed that adjustment of pH and oxidation agent amount, increase of connection time between oxidation and bioremediation, introduction of appropriate additive were capable of reducing the negative impact on soil by remediation.

Keywords: Permanganate Oxidation, Bioremediation, Petroleum Hydrocarbons, Soil Function, Soil Remediation

1. Introduction

The combined technology of potassium permanganate oxidation and bioremediation adheres to the advantages of rapid chemical oxidation and effective remediation while exhibiting a relatively mild and less environmentally destructive nature, making it a green and efficient remediation technique for petroleum hydrocarbon (TPH) contaminated soils [1, 2]. Current research on this technology has mainly focused on investigating the factors affecting its remediation effectiveness. These factors include revealing the impact of contamination range, climate, site conditions, soil properties, and remediation indicators on removal rates.

However, there has been relatively limited research regarding the preservation of native soil ecology and environmental functions. Some scholars have explored the use of biochar as an amendment and remediation agent to reduce secondary pollution [3], but research on the destruction of soil functionality and its inhibition during the remediation process remains insufficient [4, 5].

The term "soil function" refers to the maintenance of soil biodiversity, water storage and filtration, toxin degradation, and self-purification capabilities [6, 7]. These functions can be reflected in indicators such as soil organic matter (OM), dissolved organic carbon (DOC), available nitrogen and phosphorus [6]. The impact of potassium permanganate

oxidation-bioremediation on soil function can be divided into three stages: the oxidation stage, the transition stage, and the bioremediation stage. In the first stage, the (combined) reagents often interact with OM before degrading organic pollutants [8]. In the second stage, the damage caused by the oxidation treatment to the soil's microbial community makes it difficult for subsequent bioremediation to be effectively integrated. In the third stage, the intermediate products resulting from incomplete degradation during bioremediation can lead to soil toxicity issues [6]. The extent of soil damage caused by chemical oxidation [9] or oxidation-bioremediation [10] is mainly determined by their impact on soil microorganisms [11], and improvements in oxidation methods and biostimulation have been proposed as relevant measures [12]. Therefore, it is necessary to focus on the comprehensive remediation of critical stages and delve into understanding the impact of remediation actions on the soil while establishing green remediation parameters for pollutant removal and soil function recovery.

To further investigate the effects of the remediation process on the removal of soil pollutants and function, this study examines the effects and influencing factors of potassium permanganate oxidation, as well as the oxidation, transition, and bioremediation stages, on TPH and function in the soil system, with the aim of reducing the impact of the remediation process on soil function.

2. Materials and Methods

2.1. Experimental Reagents and Instruments

1. Experimental Reagents

Potassium Permanganate and Composite Reagents: A 0.2 mol/L KMnO_4 stock solution was prepared and gradually diluted. The formula for the potassium permanganate composite reagent was provided by a collaborative water plant, and it was prepared by gradient dilution. The mass

ratios of the various components were as follows: potassium permanganate (88.4%), calcium hydroxide (0.4%), sodium dihydrogen phosphate (4.3%), iron chloride (1.5%), sodium hypochlorite (0.4%), sodium silicate (0.4%), copper sulfate (0.1%), ferrous sulfate (0.1%), potassium persulfate (0.1%), acetic acid (0.1%), potassium ferrate (0.1%), polyacrylamide (1.6%), ferric chloride (1.6%), and aluminum sulfate (0.9%). The stock solution had a molar concentration of 0.2 mol/L, and the liquid concentration was also 0.2 mol/L.

Bioremediation Bacterial Agent: Bacterial strains were selected by the research team, and they possessed the capability to simultaneously remove TPH and nitrate. The selection and cultivation of these strains followed the procedures outlined in a previous publication [13].

Additives: Rhamnolipid (purity 95%), Tween-80, and sodium dodecyl benzene sulfonate (SDBS) (purity 99%) were purchased from China National Pharmaceutical Group and the 1688 platform.

2. Experimental Instruments

Total Organic Carbon Analyzer (TOC-1020A), UV-Visible Spectrophotometer (UV4802S), Gas Chromatograph (Agilent 7890B), Centrifuge (TD5), Constant Temperature Incubator (SPX-250B-Z), Shaker (HY-5A).

2.2. Preparation of Contaminated Soil

Clean soil was air-dried, sieved through a 10-mesh sieve after impurity removal, and then transferred to brown glass wide-mouth bottles for light-protected storage. The basic physicochemical properties of the clean soil are provided in Table 1. Commercially purchased No. 0 diesel fuel was dissolved in petroleum ether and poured into the wide-mouth bottles containing the soil. The solution was stirred while dissolving and then left to stand in a fume hood until all the solvent had evaporated. The contaminated soil was sealed and kept in the dark for aging for 14 days.

Table 1. Basic Characteristics of the Soil Samples.

Soil Texture	pH	Moisture Content (%)	Density (g/cm^3)	Bulk Density (kN/m^3)	Particle Density	Organic Matter Content (g/kg)
Ashy Yellow Clay	7.1	26.4	1.93	19.1	2.73	7.47

2.3. Analysis Methods

TPH (Total Petroleum Hydrocarbons) Analysis: TPH in the soil was determined using the "Gas Chromatographic Method for the Determination of Petroleum Hydrocarbons (C_{10} - C_{40}) in Soils and Sediments" (HJ 1021—2019).

Organic Matter (OM) Analysis: The organic matter content in the soil was measured according to "Soil Testing Part 6: Determination of Soil Organic Matter" (NY/T 1121.6—2006).

KMnO_4 Analysis: KMnO_4 concentrations were measured using a UV-Visible Spectrophotometer.

Alkaline Nitrogen and Available Phosphorus Analysis: The determination of alkaline nitrogen followed the

guidelines in "Determination of Soil Alkaline Nitrogen" (DB51/T 1875—2014), and available phosphorus was determined according to "Soil Testing Part 7: Determination of Soil Available Phosphorus" (NY/T 1121.7—2014).

DOC (Dissolved Organic Carbon) Analysis: For DOC analysis in soil, 10 g of soil samples were mixed with water at a 1:5 ratio, shaken at 25°C at 250 revolutions per minute for 1 hour, then centrifuged at 15,000 revolutions per minute for 10 minutes. The supernatant was filtered through a 0.45 μm membrane, and the organic carbon content was measured.

2.4. Residual Oxidant and Oxidation Experiments

Residual Oxidant Experiment: In 50 mL centrifuge tubes,

10 g of contaminated soil was mixed with 10 mL of 50 mmol/L KMnO_4 solution. The mixture was agitated at 120 rpm for 1 hour, 6 hours, 12 hours, 24 hours, 3 days, and 7 days, followed by centrifugation. The supernatant was filtered through a 0.45 μm membrane, and KMnO_4 concentrations were measured. Each group was performed in duplicate.

Oxidation Experiment: 10 g of contaminated soil was placed in 50 mL centrifuge tubes and mixed with 10 mL of varying concentrations of KMnO_4 or composite reagents. After agitation at 120 rpm, the supernatant was separated, and TPH in the soil was measured. Each group was performed in duplicate. A total of 37 batches were designed, with reaction times of 1 hour, 6 hours, 12 hours, and 24 hours. Oxidation agents used included 0.01 mol/L, 0.02 mol/L, 0.05 mol/L, and 0.1 mol/L KMnO_4 , as well as 0.05 mol/L composite reagent. Control experiments with deionized water were conducted, and reaction temperatures were maintained at 20°C and 35°C. pH levels were adjusted to 5.50, 7.50, and 9.50 by adding nitric acid and sodium hydroxide.

2.5. Potassium Permanganate Oxidation-Bioremediation of Polluted Soil and Soil Impact Experiments

100 g of polluted soil was measured into a 250 mL polytetrafluoroethylene bottle. The experiment was divided into 14 batches, with each batch conducted in duplicate. The first stage controlled the type and quantity of potassium permanganate oxidation agents. The second stage controlled the bioremediation integration time. In the third stage, different bacterial liquid concentrations and reaction times were used. Changes in soil pH, nitrogen, phosphorus, density, OM, DOC, and TPH were investigated. The oxidation time was set to 12 hours, and 150 mL of bacterial liquid was added. Deionized water was added as a blank control for bioremediation. The reaction was carried out at a constant temperature of 30°C in a shaking incubator. After the reaction, the soil was centrifuged at 3000 rpm for 10 minutes, the supernatant was removed, and the soil was then tested. Details of the experimental design can be found in Table 2.

Table 2. Experimental Design of the Effect on Soil Function during Permanganate Pre-oxidation- Bioremediation.

Sample	Oxidation	Concentration of oxidizer / (mol/L)	Integration time/(d)	Bioremediation	Bacterial liquid cell count /(cfu/L)	* Bioremediation time /(d)
1#	×	-	-	×	-	-
2#	×	-	-	√	7×10^{14}	10
3#	√	0.05	0	×	-	-
4#	√	0.05	0	√	7×10^{14}	10
5#	√	0.05	3	√	7×10^{14}	10
6#	√	0.05	7	√	7×10^{14}	10
7#	√	0.05	14	√	7×10^{14}	10
8#	√	0.05	28	√	7×10^{14}	10
9#	√	0.05	60	√	7×10^{14}	10
10#	√	0.05	90	√	7×10^{14}	10
11#	√	0.02	28	√	7×10^{14}	10
12#	√	0.1	28	√	7×10^{14}	10
13#	√	0.05	28	√	3.5×10^{14}	5
14#	√	0.05	28	√	3.5×10^{14}	10

*Design basis: The bacterial agents have a relatively short adaptation period of 24 hours for petroleum hydrocarbon intake, and the initial 5 days of the reaction belong to the rapid degradation stage (previously unpublished results).

2.6. Impact of Additives on Soil TPH and Function

To further enhance the removal efficiency of pollutants during the remediation and reduce the loss of DOC (i.e., minimizing soil function disruption), a selection study was conducted on high organic pollutant-low soil organic matter affinity additives. The evaluation focused on three aspects: the impact of additives on the consumption of oxidants in the contaminated system, their enhancement on solubilization of TPH pollutants, and their influence on DOC content.

Specifically, 15g of contaminated soil was transferred to a 50 mL centrifuge tube, and 20 mL of 0.2 mol/L KMnO_4 solution was added, followed by mixing and a 4-hour period of settling. The purpose was to evaluate the additive's effect on the consumption of the oxidant. Subsequently, 30 mL of four different additives (a. pure water, b. rhamnolipid, c.

Tween-80, d. SDBS) at a concentration of 1000 mg/L was introduced into the system, and the reaction was conducted at 200 rpm on a shaker. After 0.5 hours, 1 hour, 3 hours, 6 hours, and 12 hours, the supernatant was collected following centrifugation, and the concentrations of KMnO_4 , TPH, and TOC were measured. A parallel control group was also set up. Blank tests were conducted simultaneously for additives b, c, and d.

Because the soil is focused as the subject, the aqueous phase is removed along with solid-liquid separation. The mass of material introduced into the contaminated soil remediation system via additives, minus the mass of material discharged from the remediation system through centrifugation (ΔTPH , ΔTOC), is taken as the subject of investigation. The specific calculation methods are as follows: $\Delta\text{TPH} = 0 - \text{TPH}_{(\text{centrifugate})}$, $\Delta\text{TOC} = \text{TOC}_{(\text{additive})} - \text{TOC}_{(\text{centrifugate})}$.

3. Results and Discussion

3.1. Oxidation of TPH in Soil Using Permanganate

The results of the residual oxidant test are presented in Table 3. After 1 hour of reaction, the KMnO_4 concentration in the system remained relatively high. Over the next 1 to 6 hours, its concentration rapidly decreased, and by around 15 hours, it was nearly entirely consumed. After 24 hours, the KMnO_4 was completely depleted.

Table 3. Residual Quantity of the Permanganate during the Oxidation Process of TPH Contaminated Soil.

Reaction time	Absorbance (525nm)	c/(mmol/L)	Average
1h	0.155	1.389	1.303
	0.136	1.218	
6h	0.292	0.654	0.766
	0.392	0.878	
15h	0.003	0.001	0.003
	0.011	0.005	
24h	0.003	0.001	0.002
	0.005	0.002	

The residual TPH concentrations in different oxidation systems are illustrated in Figure 1. All experimental groups exhibited TPH removal. During the agitation reaction, TPH in the soil hardly migrated to the water, possibly related to the aging during the preparation of contaminated soil [14]. TPH removal rates were higher for 0.05 and 0.1 mol/L KMnO_4 , while the composite permanganate showed a smaller synergistic removal effect on petroleum hydrocarbons. The effect of different temperatures on TPH removal was not significantly different. TPH removal rates were higher under weak acid and weak alkaline conditions, with the highest removal rate observed under acidic conditions because manganese has a higher redox potential at this pH [15]. In alkaline conditions, the oxygen generated by sodium hydroxide also had a certain degrading and promoting effect on TPH. It was concluded that the TPH removal rate in the first stage of permanganate oxidation ranged from 18% to 61%.

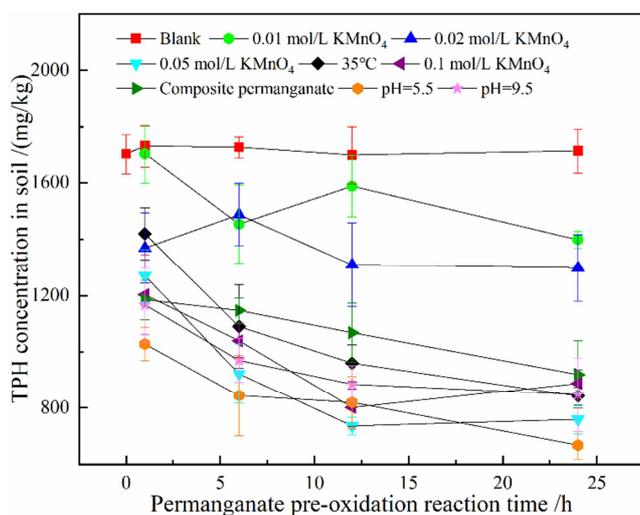


Figure 1. Effect of Permanganate Pre-oxidation on TPH Contaminated Soil.

3.2. Impact of Three Stages of Permanganate Oxidation-Bioremediation on Soil TPH and Function

The content of alkaline nitrogen and available phosphorus in contaminated soil before and after the reaction is shown in Figure 2(A), while organic matter (OM) and dissolved organic carbon (DOC) are depicted in Figure 2(B).

DOC is a direct indicator of soil ecological function quality [16]. Without oxidation and only with bioremediation, the DOC content increased from 38.2 mg/L to 73 mg/L within 10 days, likely due to the introduction of bioremediation agents. In contrast, in systems subjected to oxidation alone, the DOC content was 15.9 mg/L, significantly lower.

With prolonged bioremediation time after oxidation, the DOC concentration gradually increased, with a relatively gradual rise over the first 28 days. After 60 days, the concentration increased from 96.9 mg/L to 264 mg/L, marking a growth rate of 172%. Following this peak, there was a slight decline in DOC concentration, with the peak concentration occurring between 60 and 90 days.

As the molar concentration of KMnO_4 increased from 0.02 mol/L to 0.1 mol/L, the soil's DOC mass concentration decreased from 73.6 mg/L to 25 mg/L, a 66% reduction. This indicates that KMnO_4 has a significant impact on the system's DOC concentration. Both the concentration of the bioremediation agent and the remediation time also influenced the soil's DOC. Overall, lower bacterial liquid concentration and reduced bioremediation time led to lower DOC concentrations, consistent with the findings of the control group experiments.

OM content ranged from 6.0 to 9.2 g/kg, with the concentration in the control group systems following the order of oxidation alone < no remediation < bioremediation alone. The trend of OM changes was similar to DOC, suggesting a connection to the addition of organic matter in the bioremediation agent. Additionally, KMnO_4 had the effect of reducing soil OM. With the extension of the second-stage bioremediation time, OM content initially increased slightly and then gradually decreased. This trend might be due to the loss of organic material from the bioremediation agent during this process. Considering the simultaneous increase in DOC content, it can be inferred that there was a significant increase in the proportion of active or easily migratable organic substances within the OM component. Moreover, it was noted that high-concentration oxidizing agents had a strong reducing effect on OM.

The overall concentration of available phosphorus in the soil remained relatively constant, ranging from 0.061% to 0.087%. The combined remediation process had minimal impact on soil available phosphorus. The concentration of alkaline nitrogen in the control group systems followed the order of oxidation alone < complete control < bioremediation alone. This is likely linked to the nitrogen added through the bioremediation agent, and KMnO_4 also decreased the alkaline nitrogen concentration in the system. The concentration of alkaline nitrogen during the second stage of

the bioremediation remained generally between 0.13% and 0.148%, with a minimal effect of bioremediation time.

The numerical changes in soil pH and density remained

within 10% and were minimally affected by the combined remediation.

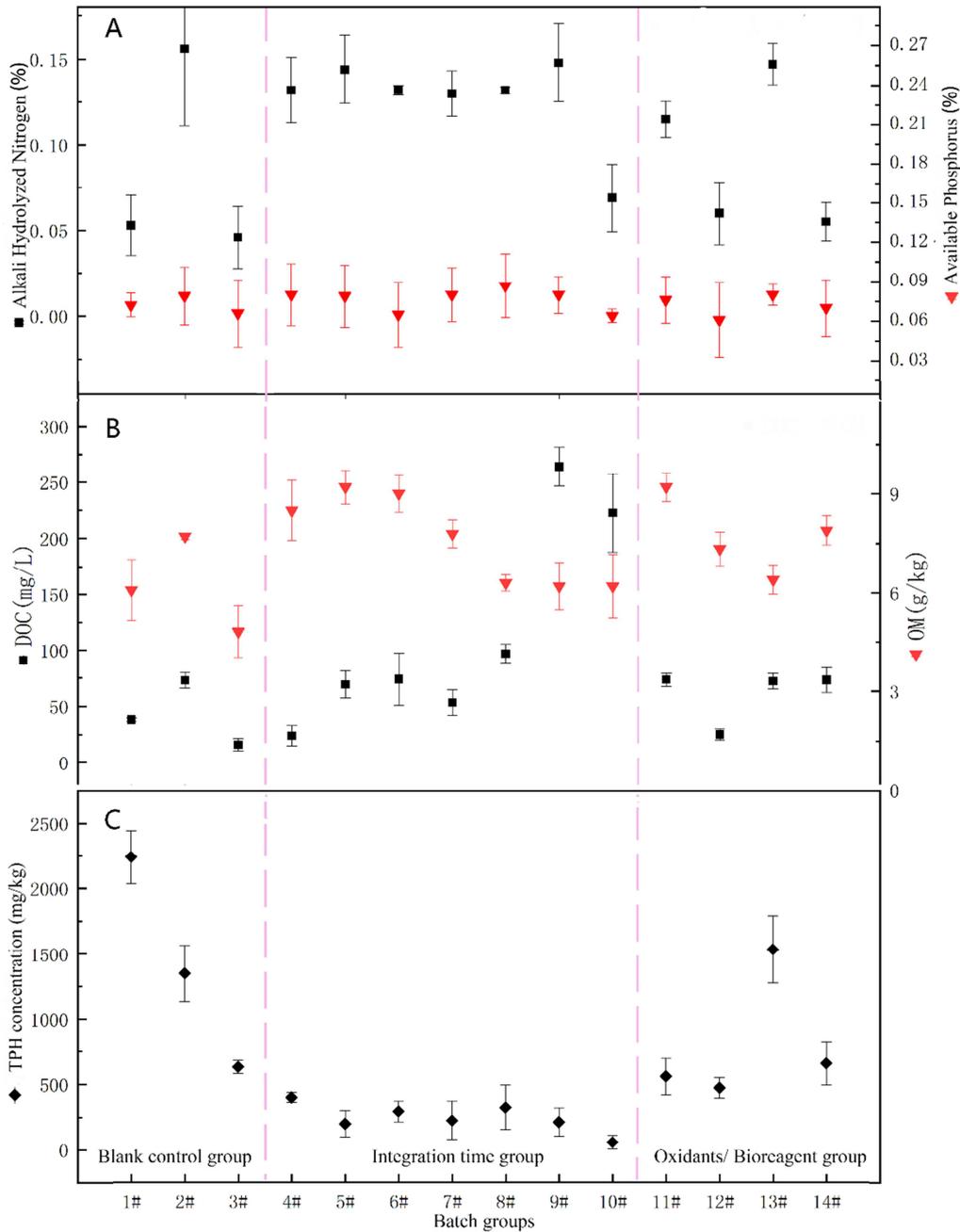


Figure 2. Effect of the Three Phases of Permanganate Pre-oxidation – Bioremediation on Soil. (A) Alkali Hydrolyzed Nitrogen and Available Phosphorus, (B) DOC and OM, (C) TPH.

The concentration of TPH in contaminated soil before and after the reaction is shown in Figure 2(C). Compared to the control group at 2240 mg/kg, TPH concentration decreased to 1352 mg/kg in the only bioremediation group, a decrease of 39.6%. TPH concentration decreased to 632 mg/kg in the only oxidation group, marking a reduction of 71.8%. The oxidant's impact on TPH removal was stronger than that of the bioremediation agent. $KMnO_4$ consumed organic matter on the soil surface, causing desorption [17] and migration of

TPH from organic matter into the aqueous phase, leading to its removal.

During the second stage, from 0 to 60 days, the mass concentration of TPH fluctuated within the range of 200 to 424 mg/kg. While bioremediation time had a direct impact on ecological parameters such as soil DOC, its effect on TPH removal was relatively weaker. Even after 5 days of bioremediation, the TPH concentration in the system remained relatively high. However, after 10 days, there was a significant

reduction in TPH mass concentration, dropping to 692.8 mg/kg. Doubling the concentration of the bioremediation agent reduced TPH concentration from 659.2 mg/kg to 324 mg/kg. Both the bioremediation agent's concentration and reaction time played a role in reducing TPH, with reaction time having a more significant impact. Following oxidation and around 10 days of bioremediation, the TPH removal rate in the contaminated system stabilized at over 70%. With a bioremediation time exceeding 60 days, the removal rate

reached over 90%, with the highest removal rate at 97.35%.

A comparison of TPH concentrations before and after each segment is shown in Table 4. The changes in aliphatic hydrocarbons and aromatic hydrocarbons were similar. The concentrations of the C₁₀-C₁₂ segment decreased significantly, nearly disappearing, and the C₁₃-C₁₆ segment decreased by approximately 60%. The C₁₉-C₄₀ segments each saw a reduction of about 40%. The removal rate of high-carbon chain segments remained acceptable.

Table 4. Mass Concentration Distribution of TPH Segmentation in Soil Before and After the Remediation.

Hydrocarbon Type		Concentration Before Reaction/mg/kg	Concentration After Reaction/mg/kg
Aliphatic	C ₁₀ -C ₁₂	167	6
	C ₁₃ -C ₁₆	619	252
	C ₁₇ -C ₂₁	668	388
	C ₂₂ -C ₄₀	170	90
	C ₁₀ -C ₁₂	44	2
Aromatic	C ₁₃ -C ₁₆	185	74
	C ₁₇ -C ₂₁	147	93
	C ₂₂ -C ₄₀	28	17

3.3. Impact of Additives on KMnO₄, OM, and TPH

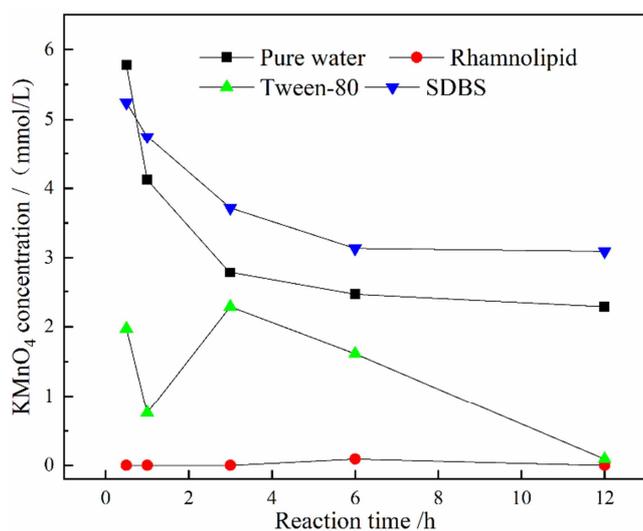


Figure 3. Molar Concentration of Potassium Permanganate in Soil Supernatant on Various Treatment Time in Additive Groups.

The concentration of KMnO₄ in the soil supernatant at various reaction time is shown in Figure 3. Using distilled water as a reference, it was observed that SDBS had a solubilizing effect on residual KMnO₄ in the soil. On the other hand, Tween-80 and rhamnolipid showed weaker solubilizing effects. For rhamnolipid group, only a very low concentration of KMnO₄ was detected in the aqueous phase at 6 hours into the reaction, indicating that it would not significantly cause residual oxidant migration to the aqueous phase. This could be attributed to the high organic content in rhamnolipid, which reacted quickly and had good water solubility, making it prone to oxidation and rapidly consuming KMnO₄. As for Tween-80, it increased in viscosity upon contact with water, formed more foam, and

dissolved slowly. In the later stages of the reaction, the rate of KMnO₄ concentration decrease gradually increased [18]. Although SDBS contained organic components, its impact on KMnO₄ consumption was relatively minimal.

Changes in Δ TOC and Δ TPH concentrations are shown in Figures 4 (A) and (B), respectively. Negative Δ TOC values indicate that additives lead to the loss of TOC. Tween-80 significantly increased the system's TOC, and it remained stable within the first 12 hours. In contrast, the addition of pure water and rhamnolipid substantially reduced the TOC. On the other hand, Tween-80 significantly promoted the migration of TPH from the soil to the aqueous phase, with Δ TPH stabilizing at over 2 mg/L and increasing with reaction time. Pure water, SDBS, and rhamnolipid had a smaller solubilizing effect on TPH. In the pure water system, the solubilization of TPH increased with reaction time, following typical dissolution characteristics. rhamnolipid showed the least solubilizing effect on TPH, possibly due to its high content of active organic components, enhancing the adsorption of TPH on rhamnolipid and the soil surface [19].

Combining Figures 3 and 4, it can be concluded that Tween-80, while increasing the system's TOC, has a strong solubilizing effect on TPH and leads to the greatest migration to the aqueous phase. This promotes the presence of active organic components in the soil system and results in effective TPH removal. Additionally, Tween-80 exhibits a slow consumption of oxidants in the early stages of the reaction, making it an ideal high-organic-pollutant, low-soil-organic-matter-affinity additive. SDBS has very low consumption of oxidants, can introduce active organic materials, and has a reasonable solubilizing effect on TPH. In contrast, pure water and rhamnolipid have a poorer impact on soil protection and function.

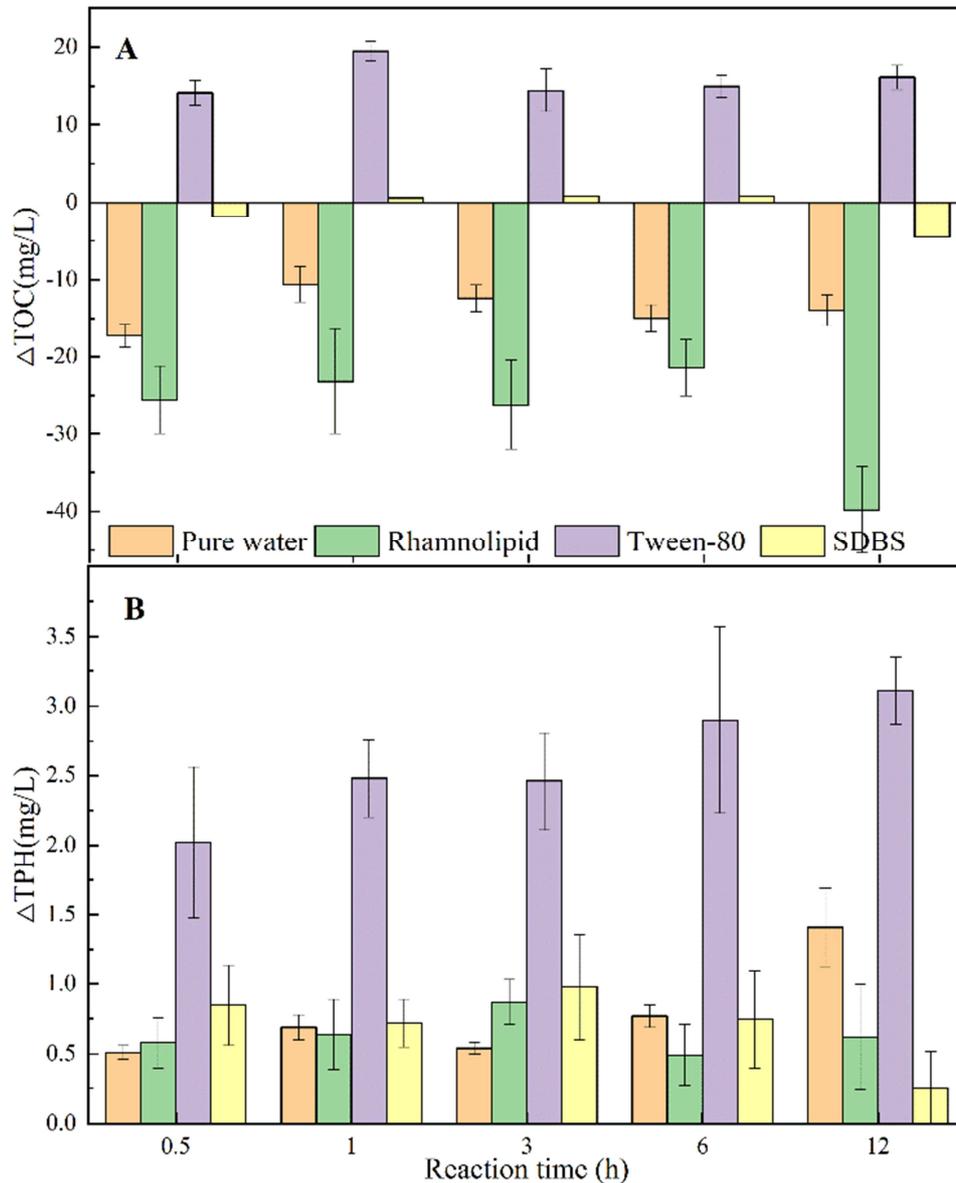


Figure 4. Variation of Mass Concentration of the Parameters in the Supernatant and the Additive, (A) Δ TOC, (B) Δ TPH.

4. Conclusion

In terms of pollution reduction, the complete consumption time of KMnO_4 is approximately 24 hours. KMnO_4 concentrations of 0.05 and 0.1 mol/L, as well as acidic conditions, exhibit strong TPH removal capabilities, while the influence of composite agents and temperature is comparatively weaker. Bioremediation shows slightly lower efficacy in TPH removal than the oxidants, but combined remediation achieves a total TPH removal rate exceeding 70%, with the highest rate reaching 97.35%. Regarding the impact on soil function, KMnO_4 is associated with substantial soil damage. With reduced microbial concentration and shorter remediation time, soil DOC concentrations decline. However, as the recovery time increases, both DOC concentrations and the proportion of active organic matter within the soil show significant

improvements. Furthermore, the influence of additives is notable, particularly Tween-80, which enhances TOC within the system, exhibits the highest solubilization effect on TPH, and provides the best removal efficiency for petroleum hydrocarbon pollutants. Moreover, it exhibits a slower consumption of oxidants in the early stages, making it a relatively ideal additive for high-organic-pollutant, low-soil-organic-matter-affinity scenarios. By adjusting pH to a weakly acidic state, oxidant concentrations, increasing the connection time between oxidation and bioremediation, and introducing Tween-80 and other suitable additives, the impact of remediation on soil function can be reduced.

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Conflicts of Interest

The authors declare no conflicts of interest.

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