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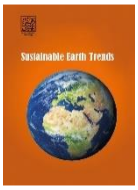

Low-temperature thermal desorption of phenanthrene and pyrene from oil-contaminated soil: modeling of removal using response surface methodology

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ABSTRACT

Petroleum hydrocarbons are toxic to humans and other organisms, and some are carcinogenic. Various remediation techniques have been developed to address this issue sustainably, including thermal methods. Low-temperature thermal desorption (LTTD) is a treatment technology that offers an environmentally sustainable approach to removing contaminants from solid media by volatilizing them with heat but without combustion of the media. This study is aimed at evaluating the efficiency of LTTD to remove phenanthrene and pyrene, and their mixture from contaminated soils. The soil samples were contaminated with these compounds at three concentration levels, including 2000, 4000, and 8000 mg kg^{-1} at different temperatures (i.e., 100, 200, and 300 °C) and retention times (i.e., 30, 60, and 90 min). The compounds were extracted using an ultrasonic bath and analyzed with HPLC. Analysis of the findings obtained by the Taguchi method indicated that increased concentration, temperature, and retention time led to a higher efficiency in removing the compounds from contaminated soils. The results indicate that increasing temperature, retention time, and contaminant concentration positively influence removal efficiency, with the highest desorption rates reaching over 88%. These findings suggest that LTTD is a promising solution for addressing soil contamination by petroleum hydrocarbons, offering an efficient and eco-friendly approach for remediation. Therefore, LTTD could be a highly effective and sustainable solution for removing pollutants from contaminated soil, as well as in severe contamination.

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1. Introduction

Crude Oil compounds are the most important organic pollutants in the environment, particularly in soil. Their entry into nature has become one of the conservationists' main concerns because of their toxicity and carcinogenicity. However, these pollutants are highly stable in soil, and their gradual accumulation in soil over time causes disturbance in the normal functioning of soil (Zhao et al., 2019). Contamination of soil with crude oil may occur during its extraction, transportation, or refining. Contamination could lead to damage to the environment, the plant, and the animal population. Public concerns relevant to toxic organic compounds such as crude oil hydrocarbons, particularly polycyclic aromatic hydrocarbons, entering the environment widely are increasing. These compounds are considered organic pollutants that are resistant to biological decomposition and could bring about adverse environmental effects (Arbabi, 2009). Today, the teratogenicity and mutagenicity of PAHs have been proven (Rashid et al., 2009). They consist of two or more fused aromatic rings that exist as a variety of isomers. They are present mainly as a complicated mixture in the environment (Sörengård et al., 2020). Their sources include: forest fires, volcanic activity, incomplete combustion of fossil fuels, etc. They can be dispersed in the environment at different concentrations (Cripps et al., 1990). Typically, PAHs found in plants and aquatic animals are much more than those in water or soil due to their aggregation properties (Anyakora et al., 2011). Phenanthrene is a tricyclic aromatic compound, while Pyrene contains four aromatic rings. These are among the leading pollutants according to the US Environmental Protection Agency (EPA) (Bento et al., 2005). Given the persistence and toxicity of these pollutants, sustainable remediation strategies are essential to mitigate their environmental impact.

Different approaches like non-ionized surfactants (Chi., 2011), biological inoculation (Bento et al., 2005), electrical oxidation (Tsai et al., 2010), and chemical oxidation (Do et al., 2009) have been used to remove these compounds. Among these approaches, heat treatment procedures are more popular and commonplace compared to other approaches due to the high efficiency, low cost, and time required (Falciglia et al., 2011). Heat treatment is divided into two categories: thermal desorption and thermal destruction. Thermal desorption procedures are further classified into low-temperature thermal desorption (LTTD) conducted within 90-320°C and high-temperature thermal desorption (HTTD) conducted within 320-550°C (Merino and Bucalá, 2007).

Thermal desorption is a physical process in which organic compounds are evaporated by heat and separated from the contaminated soil. As a more sustainable approach compared to other remediation techniques, it minimizes the use of chemical reagents and reduces long-term soil contamination risks. In fact, the pollutants are transferred from one phase to another throughout this process (Falciglia et al., 2011). Recent studies have explored various aspects of thermal desorption for the removal of polycyclic aromatic hydrocarbons (PAHs) from contaminated soils. Zhang et al. (2024) investigated thermal desorption processes for pyrene removal and developed an optimized model to predict the efficiency of this process. Their findings indicated that temperature and residence time significantly affect the desorption efficiency. In another study, Zhang et al. (2024) utilized machine learning techniques to predict the thermal desorption efficiency of PAHs in contaminated soils. This study demonstrated that machine learning models could accurately predict the outcomes of thermal treatment based on temperature and time factors. Additionally, Zhang et al. (2024) examined the influence of temperature and the water-to-organic matter ratio on pyrene removal, revealing that adjusting temperature and moisture levels could significantly enhance the removal efficiency. In a further investigation, Zhang et al. (2024) assessed the impact of temperature and residence time on the physical and chemical properties of soils post-treatment, highlighting that these parameters directly influenced the soil structure. Finally, Zhang et al. (2024) introduced various numerical models for optimizing thermal treatment conditions to enhance PAH removal, demonstrating that the application of these models could improve process efficiency.

This study aims to evaluate the efficiency of low-temperature thermal desorption, a sustainable remediation technique (Song et al., 2018), in removing phenanthrene, pyrene, and their mixture within different retention intervals and at different temperatures. In the present work, low-temperature thermal desorption treatment of aromatic-contaminated soils was studied using an experimental bench-scale apparatus. The main goals of the work were: (i) to test a bench-scale apparatus in order to predict optimum conditions for thermal treatment of organic contaminant polluted soils; (ii) to assess the effect of temperature on the soil adsorption capability; (iii) to assess the influence of contact time and different concentration of aromatic hydrocarbons on contamination removal efficiency; (iv) to model the experimental data in order to calculate the desorption parameters needed to optimize the treatment operating conditions and to guide the design and the scale-up of low-temperature desorption systems. The primary objective of this study is to explore the effectiveness of low-temperature thermal desorption (LTTD) in removing hazardous polycyclic aromatic hydrocarbons (PAHs) such as phenanthrene and pyrene from contaminated soils. The novelty of this approach lies in its ability to achieve high removal efficiencies without the need for harmful chemical reagents, positioning LTTD as a sustainable and cost-effective alternative for soil decontamination in industrial and environmental applications.

2. Material and methods

2.1. Sample preparation

Samples of soil free from coarse sand and gravel were obtained. To control potential intervention due to the possible existence of organic compounds in the samples, the samples were heated in a furnace at 500°C. These samples were artificially contaminated with phenanthrene (tricyclic) and pyrene (tetracyclic) at concentration levels of 2000, 4000, and 8000 mg kg^{-1}. In this experiment, approximately 300g of the soil samples contaminated with phenanthrene (Phe.), pyrene (Pyr.), and a combination of both were tested within three intervals (30, 60, and 90 min) and at three different temperatures (100, 200, and 300°C) using a 50-500°C laboratory oven. To control the accuracy of oven temperature and calibration, a special thermometer with high-temperature tolerance was used. All steps were conducted at laboratory conditions. A total of 81 samples were examined. Phenanthrene ($C_{14}H_{10}$, *purity* > 97%) and pyrene ($C_{16}H_{10}$, *purity* > 97%) were purchased from Merck Co. (Germany). Analysis of soil graining was done per Unified Soil Classification System (USCS) (ASTM-d-2478). By this method, the soil sample was passed through a stack of sieves from mesh #4 (pore size 4.75 mm) to mesh # 200 (pore size 0.075 mm). In grain analysis, 300-500g of soil was used in each test (Arbabi et al., 2004).

2.1.1. Analysis, extraction, and cleanup of samples

In this study, the ultrasonic method was used to extract oil compounds from contaminated samples. 2g of soil was weighed and poured into the tube, and then 5 mL of acetonitrile was added to the soil. The solution was placed in a stainless steel (CD-4820) ultrasonic bath, 170 W, 250 × 100 × 60 mm dimensions for 60 minutes at 50-60°C. After cleanup and sample preparation, HPLC analysis was done according to EPA and NOISH. The Agilent HPLC equipment used was equipped with a degasser and a UV detector. The C18 column used had a diameter of 4.6 mm and a length of 150 mm. The mobile phase was water/acetone at a flow rate of 1.5 µl min^{-1}. Concentration and the pollutant type were selected as variables, each at three levels. The selected levels for the variable are according to the Taguchi method (Table 1). Optimum conditions for determining phenanthrene and pyrene concentrations using HPLC are shown in Table 2. In order to analyze and standardize phenanthrene and pyrene by HPLC, we used the series dilution method. Analysis characteristics and standard amounts of phenanthrene and pyrene based on series dilution methods are depicted in Table 3. Calibration curves for phenanthrene and pyrene using HPLC are depicted in Fig. 1.

Table 1. Investigating parameters and their levels in optimization.

Parameter	Levels		
	1	2	3
Temperature (°C)	100	200	300
Time (min)	30	60	90
Concentration (mg kg^{-1})	2000	4000	8000
Type of hydrocarbon	Phe.	Pyr.	Mixture of Phe. and Pyr.

Table 2. Optimum conditions for determining the phenanthrene and pyrene concentrations by HPLC.

Items	Values
Flow rate (mL/min)	1.5
Operating time (min)	20
Retention time (min)	7.5±0.2 for phenanthrene 10.5±0.4 for pyrene
Mobile phase	Acetonitrile/water (40/60 v/v)
Wave length	254 nm
Temperature (°C)	20
Pressure (bar)	120

Table 3. Series dilution method and analysis conditions of phenanthrene and pyrene by HPLC

Standard no.	Phe. Standard concentration (ng/μg)	Pyr. Standard concentration (ng/μg)
1	6.25	6.25
2	12.5	12.5
3	25	25
4	50	50
5	100	100
Formula	$C_{14}H_{10}$	$C_{16}H_{10}$
Molecular weight	178	202
No. of benzene rings	3	4
Exit time of peak (time)	7.356±0.5	10.884±0.5
Correlation factor (R)	0.99920	0.99212
Linear equation	Y=132.80478X+143.29208	Y=41.45840X+69.80869

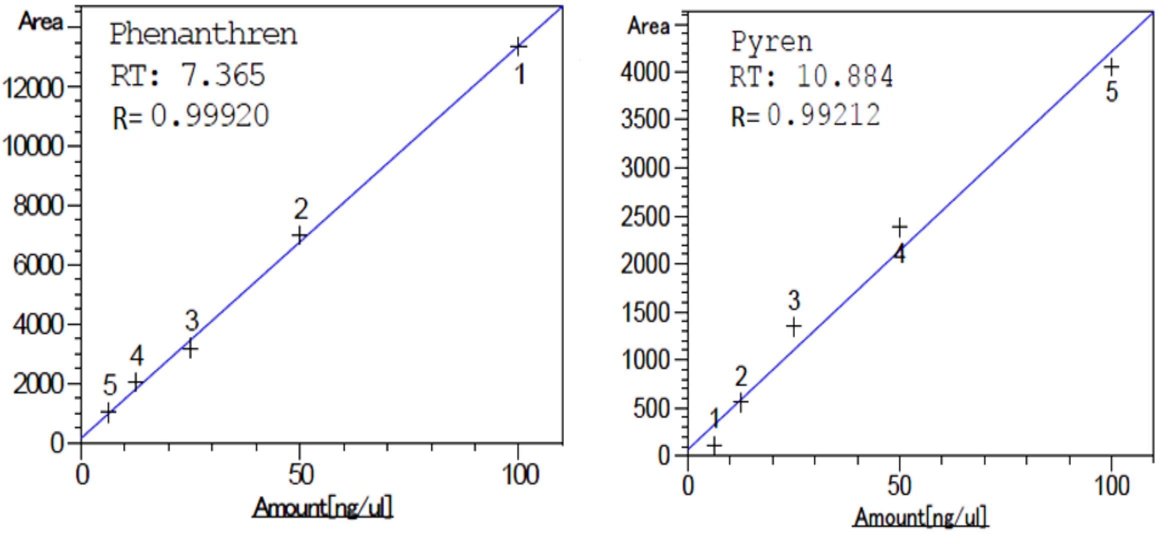

Fig. 1. Calibration curves for phenanthrene (left) and pyrene (right) using HPLC.

In the Taguchi method, a converted response function defined as the effect mark (S)/error-derived effect (N) ratio is used for a more accurate analysis of data. The advantage of using this new response, in contrast to the primary response, in data analysis is to compare effect magnitudes due to each main factor with those of the effects due to errors and confounders in measurement, leading to a more accurate interpretation of the actual effect of factors on the system (Jafarzadeh et al., 2009). S/N ratio is calculated as follows in Eq. 1:

$$\frac{S}{N} = -10log\frac{\left(\frac{1}{{y_1}^2}+\frac{1}{{y_2}^2}+\cdots+\frac{1}{{y_n}^2}\right)}{n} \qquad (1)$$

Where y_n represents the measured response in each test, and n represents the number of repeated tests (equal to 2 in this study). Here, the larger the S/N ratio, the better the condition (larger is better).

3. Results and Discussion

The obtained findings on thermal desorption of phenanthrene at 2000, 4000, and 8000 mg kg^{-1} at 100, 200, and 300°C in 30, 60, and 90 min times are shown in Fig. 2. As illustrated, the removal efficiency of phenanthrene at all concentrations increased as the temperature increased. The highest removal efficiency of phenanthrene at 2000 mg kg^{-1} at 100°C was 34% while it increased to 88.76% at

300°C. Also, the removal efficiency of phenanthrene increased as time increased, but at approximately all temperatures under study removal efficiency increased rapidly up to 60 min; however, the increasing trend became slower afterward.

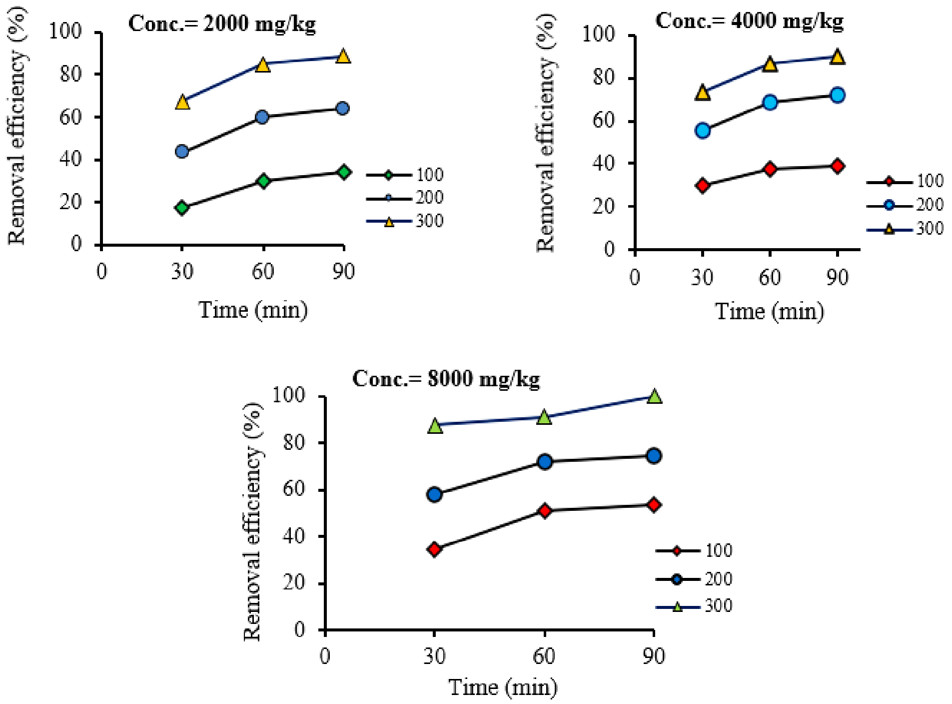

Fig. 2. Removal efficiencies of Phenanthrene in different temperatures and concentrations.

The findings on thermal desorption of pyrene are shown in Fig. 3. As illustrated, the removal efficiency of pyrene at all concentrations increased as the temperature increased. For example, the highest removal efficiency of pyrene at 2000 mg kg^{-1} at 100°C was 44.9% while it increased to 93.56% at 300°C. Also, the removal efficiency of pyrene increased as time increased, as expected. But at approximately all temperatures under study, removal efficiency increased rapidly up to 60 min and the increasing trend became slower afterwards.

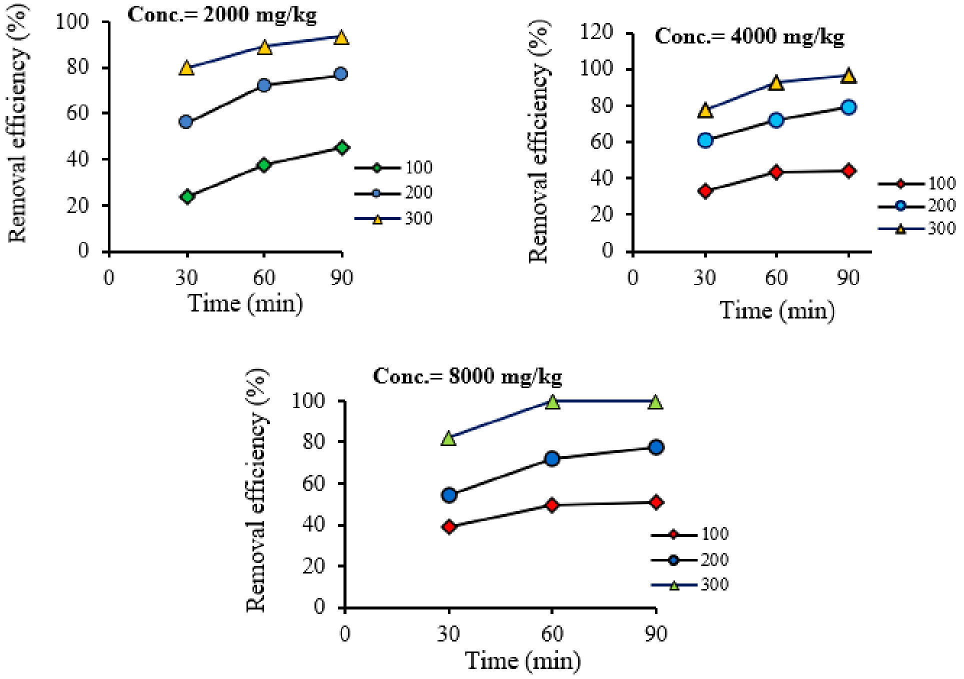

Fig. 3. Removal efficiencies of Pyrene at different temperatures and concentrations.

As Fig. 4 illustrates, the removal efficiency of phenanthrene and pyrene combination at all concentrations increased as the temperature increased. For example, the removal efficiency of phenanthrene and pyrene combination at 2000 mg kg^{-1} was registered as 40.03% at 100°C and 81.90% at 300°C. The removal efficiency of phenanthrene and pyrene combination increased as with either alone, and also increased rapidly at all temperatures under study up to 60 min, but the increasing trend became slower afterward.

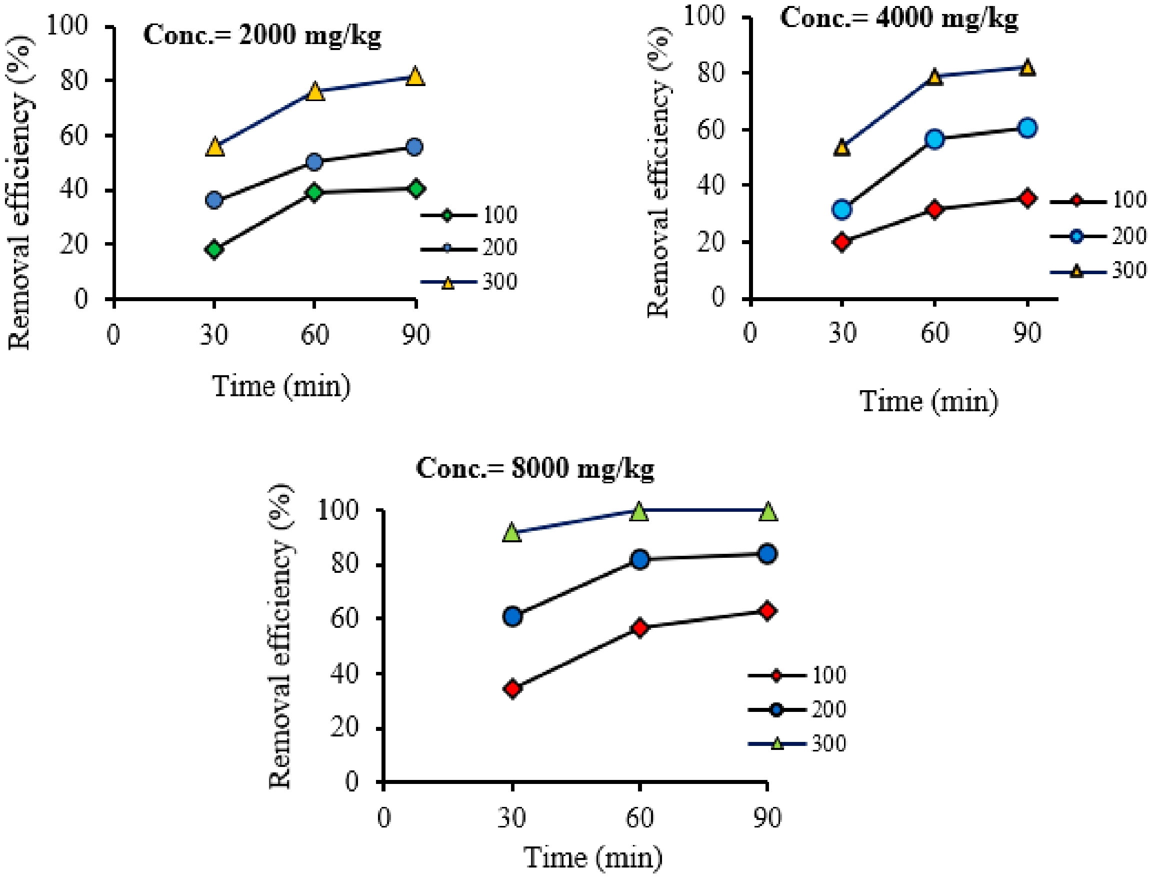

Fig. 4. Removal efficiencies of phenanthrene and pyrene combination at different temperatures and concentrations.

Analysis of the findings obtained by the Taguchi method indicates that increased concentration, temperature, and retention time led to a higher efficiency in removing the pollutants. Figs. 1 to 3 show that temperature and time are two main factors contributing to the desorption of these compounds from soil. This aligns with recent findings by Zhu et al., who reported that the addition of nanoscale zero-valent iron (nZVI) significantly improved the desorption efficiency of chlorinated hydrocarbons, achieving over 99% removal at 300 °C for 2 hours (Zhu et al., 2024). The findings of the present study are consistent with Ken and Lo's study (Ken and Lo, 2002). Lee et al. (1998) study indicated that the maximum efficiency of thermal desorption was dependent on the used temperature, so that the efficiency of thermal desorption from oil-contaminated soil was lower than 70% at 150°C temperature after a 120-min retention time while it was higher than 90% at 300°C temperature after a 30-minute retention time (Lee et al., 1998).

In this study, the efficiency in removing the pollutants under study increased rapidly at all concentrations and approximately all temperatures as retention time increased up to 60 minutes and continued to increase slowly afterward. This could be explained by the two phases occurring in heat-mediated removal procedures. In the first phase, evaporation from soil particles proceeds rapidly, and in the second phase, the evaporation declines because of internal diffusion (Keyes and Silcox, 1994). Our findings in this regard are consistent with the Falciglia et al. study (Falciglia et al., 2011). Also, Smith et al study of soil pollutants indicated that the efficiency of thermal desorption increased as the temperature and retention time increased (Smith et al., 2021).

This observation is consistent with the study by Yi Zhu et al. (2024), which noted a rapid desorption rate in the initial minutes at various temperatures, followed by a decline over time (Zhu et al., 2024).

Also, the study by Torrents *et al* on thermal desorption of aromatic compounds from active carbon at low temperatures indicated that the desorption rate is rapid within the first minutes at any temperature and then this rate declined over time (Torrents et al., 1997), which confirms the present study findings.

Although efficiency in removing the pollutants increases as temperature and retention time increase, but increases the consumed energy and consequently the operational costs, which makes it uneconomical. Therefore, conditions in terms of temperature and retention time should be obtained so that both an acceptable efficiency is achieved and the procedure becomes economical. According to the obtained findings, a combination of phenanthrene and pyrene at 4000 mg kg^{-1} concentration treated at 200 °C resulted in removal efficiencies of 31.31%, 56.41%, and 60.85% for retention times of 30, 60, and 90 minutes, respectively. Notably, the increase in removal efficiency from 60 to 90 minutes was marginal, whereas energy consumption rose significantly. Thus, adopting a 60-minute retention time could conserve energy and enhance economic feasibility.

4. Conclusion

In this study, the effectiveness of Low-Temperature Thermal Desorption (LTTD) in removing aromatic hydrocarbon contaminants from soil contaminated with phenanthrene and pyrene was evaluated. The results showed that increasing temperature, retention time, and contaminant concentration led to a higher removal efficiency. Specifically, the highest removal efficiency was observed at higher temperatures (300°C) and longer retention times (60 minutes), with removal rates exceeding 88%. The findings indicate that LTTD can be an efficient and sustainable method for remediating soils contaminated with petroleum hydrocarbons, particularly in cases of high contamination and severe pollutant concentrations. Compared to other methods, this approach does not require the use of additional chemical agents, minimizing environmental impact. However, the study also found that while removal efficiency increases with temperature and retention time, this improvement is not linear, and after 60 minutes, the removal efficiency tends to plateau. Additionally, increasing retention time beyond 60 minutes significantly increases energy consumption, which could raise operational costs. Therefore, for optimal performance in terms of efficiency and cost-effectiveness, a 60-minute retention time is recommended. Overall, this study demonstrates that LTTD could be a promising solution for removing hydrocarbon pollutants from contaminated soils. However, further research is needed to optimize conditions and reduce operational costs for large-scale industrial applications.

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