

Artículo Original

***EVALUATION OF PHYTOREMEDIATION AS AN ALTERNATIVE
FOR THE TREATMENT OF DRILLING SLUDGE
IN THE OIL INDUSTRY***

***EVALUACIÓN DE LA FITORREMEDIACIÓN COMO ALTERNATIVA PARA EL
TRATAMIENTO DEL LODO RESIDUAL DE PERFORACIÓN EN LA
INDUSTRIA PETROLERA***

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ABSTRACT

Introduction:

This research conducts a chemical characterization of drilling sludges generated in the oil industry, and evaluates the efficiency of hydrocarbon removal through phytoremediation.

Objective:

To evaluate phytoremediation as an alternative for the treatment of drilling sludge.

Materials and Methods:

Partial characterizations of the sludge and sludge-soil mixtures were performed, including pH, electrical conductivity, and total petroleum hydrocarbons. In addition, the removal of petroleum hydrocarbons in the mixtures after phytoremediation was also evaluated, as well as the adaptability of the plants.

Results and Discussion:

The results demonstrate that the concentration of total petroleum hydrocarbons is higher in the wet sludge compared to the dry one. High percentages of removal of these



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contaminants were also obtained after phytoremediation in both types of sludges. Sensory evaluation of the plants revealed a positive adaptability under the conditions of contamination to which they were subjected.

Conclusions:

By implementing the phytoremediation system over a period of 18 months, high efficiencies of petroleum hydrocarbon removal were achieved in both types of sludges. In addition, the phytoremediation plants have shown tolerance and adaptability to the presence of these pollutants. Based on the results obtained, it can be affirmed that phytoremediation can be an alternative in the treatment of mixtures of soil and residual drilling muds.

Keywords: drilling cuttings; phytoremediation; residual muds; removal.

RESUMEN

Introducción:

En esta investigación se realiza una caracterización química los lodos de perforación generados en la industria petrolera, y se evalúa la eficiencia de remoción de hidrocarburos mediante fitorremediación.

Objetivo:

Evaluar la fitorremediación como alternativa para el tratamiento del lodo residual de perforación.

Materiales y Métodos:

Se realizaron caracterizaciones parciales de los lodos y las mezclas suelo lodo, incluyendo pH, conductividad eléctrica e hidrocarburos totales del petróleo. Además, también se evaluó la remoción de hidrocarburos del petróleo en las mezclas, después de la fitorremediación, así como la adaptabilidad de las plantas.

Resultados y Discusión:

Los resultados demuestran que la concentración de hidrocarburos totales del petróleo es superior en el lodo húmedo que en el seco. También se obtuvieron altos porcentos de remoción de estos contaminantes después de la fitorremediación en ambos lodos. Al evaluar sensorialmente las plantas, se observó una capacidad de adaptación positiva bajo las condiciones de contaminación a las que han sido sometidas.

Conclusiones:

Con la implementación del sistema fitorremediador en un período de 18 meses, se obtuvieron elevadas eficiencias de remoción de hidrocarburos del petróleo en ambos lodos. Además, las plantas fitorremediadoras han mostrado tolerancia y adaptabilidad a la presencia de estos contaminantes. Atendiendo a los resultados obtenidos, se puede afirmar que la fitorremediación puede ser una alternativa en el tratamiento de mezclas de suelo y lodos residuales de perforación.

Palabras clave: cortes de perforación; fitorremediación; lodos residuales; remoción.

1. INTRODUCTION

Soil contamination is often associated with industrial activities, including the generation of waste materials such as drilling mud during the oil extraction processes (Gutsens and Rodríguez, 2023). The Empresa de Perforación y Extracción de Petróleo del Centro, a Cuban company dedicated to meeting part of the country's energy needs, aims for rational and efficient oil exploitation with environmental responsibility. It identifies these sludges as one of the main problems affecting the environment, González (2017). Drilling muds contain a large number of chemical additives that are pumped into the producing well to act as a lubricant and coolant for the drill bit, to bring suspended rock fragments to the surface, and to maintain the integrity of the well walls, among other functions. During this process, crushed rock is also generated by the drill bit and extracted from depths that can reach up to six kilometers (drilling cuttings), Torres (2014).

Drilling muds can be water-based or oil-based. Water-based muds (WBM) have barite (barium sulfate) and calcium carbonate as the main components, with the addition of inorganic compounds such as bentonite and other clays to increase viscosity. Oil-based muds (OBM) contain mineral oil with variable amounts of aromatic hydrocarbons, silt to increase pH and control corrosion, lignite-based chemicals to control fluid loss, emulsifiers, and detergents, Arévalo, (2018).

When drilling with OBM, an additional solids control system consisting of a vertical centrifuge or dryer and a horizontal centrifuge is used to extract the residual mud in the cuttings and recover it as part of the active mud system. The mud can be reused in the same well and in other wells as long as it does not lose its properties.

The process primarily generates two waste streams that are hazardous waste: contaminated cuttings from the vertical centrifuge (dry sludge) and contaminated cuttings from the horizontal centrifuge (wet sludge). All of these wastes are disposed of in open ponds in the environment. These ponds are a major source of contamination because the wastes migrate to the underground layers of the soil, and the ponds overflow when there is abundant rainfall, causing contamination of the surface water sources (Bravo, 2007; Tapia, 2021).

There are different forms of treatment for these sludges, such as thermal desorption, treatment with ultrasonic waves, use in cement factories, and use as construction material, among others, but most of them have high costs and even contribute to the degradation of the treated site (Ambaye et al., 2022).

In contrast, there are plants that can naturally establish themselves in contaminated environments and remove these contaminants, a property that is exploited in a technique called phytoremediation.

Phytoremediation is a process that relies on the interactions between plants, soil, and microorganisms to address soil contamination (Ruley et al., 2022). Phytoremediation has high potential in tropical areas because microbial degradation is promoted by high temperatures, and plant reproduction is not limited by hibernation conditions (although it may be affected by seasonal drought) (Arias, 2017; Hussein et al., 2022).

The objective of this work is to evaluate phytoremediation as an alternative for the treatment of drilling sludge.

2. MATERIALS AND METHODS

2.1 Characteristics of the phytoremediation experimental system

The experimental system (Figure 1) consisted of two phytoremediation systems, one with wet sludge-soil mixture (white box) and another with dry sludge-soil mixture (green box), both measuring 0.45 m in length, 0.33 m in width, and 0.2 m in depth, with a surface area of 0.15 m². Only the rhizomes of young *Cyperus alternifolius* plants were collected and hand-planted at an approximate distance of 0.02 m. The *Cyperus alternifolius* was chosen because it has been previously studied in our country and has shown high percentages of removal of fats, oils, and hydrocarbons, as well as high resistance to adverse conditions (Roche et al., 2016). It is a perennial, herbaceous species that thrives under stressful conditions (Cordero and Velásquez, 2023).



Figure 1. Experimental phytoremediation system. Source: Taken by the authors

Red ferralitic soil was also used as part of the substrate, which has a high mineral content that favors the removal of contaminants in the phytoremediation system, Malca (2020). Research on the treatment of domestic wastewater through phytoremediation has shown satisfactory results using red ferralitic soil (Domínguez-Martínez et al., 2019). This soil was previously characterized by Villar et al., (2011), and the results of this characterization are shown in Table 1.

Furthermore, based on several research studies, microbes and soil have a synergistic effect, and their combination can increase their ability to degrade pollutants (Aziz et al., 2024; Chen et al., 2015). Therefore, it is considered essential to ensure greater efficiency in the degradation of petroleum hydrocarbons and plant survival by starting with a higher proportion of soil in the mixtures, specifically 0.017 m³ of soil and 0.005 m³ of sludge (at a ratio of 3:1).

Table 1. Characterization of the red ferralitic soil used in the experimental phytoremediation systems

| <i>Parameters</i> | <i>Values</i> | <i>Units</i> | <i>Parameters</i> | <i>Values</i> | <i>Units</i> |
|-------------------------|---------------|--------------|-------------------|-----------------|--------------|
| pH-KCl | 5.74 ± 0.03 | - | Total phosphorus | 1649.86 ± 31.81 | mg/L |
| pH-H ₂ O | 6.81 ± 0.02 | - | Organic matter | 12.30 ± 0.50 | % |
| Electrical conductivity | 130.70 ± 3.20 | µS/cm | Total carbonate | 0.63 ± 0.32 | % |
| Total nitrogen | 4133.50±19.64 | mg/L | CIC | 18.10 ± 0.93 | meq/10 |

| | | | | | |
|--------------------|------------------------|-------|----|---------------------|-------|
| | | | | | 0g |
| N-NH ⁴⁺ | 16.94 ± 0.11 | mg/L | Cd | 10.35 ± 0.10 | mg/kg |
| Zn | 486.30 ± 34.10 | mg/kg | Cr | 132.02 ± 11.12 | mg/kg |
| Fe | 122880.00 ± 1742.30 | mg/kg | Mn | 7429.00 ± 200.80 | mg/kg |
| Ni | 251.03 ± 38.01 | mg/kg | Pb | 59.00 ± 8.20 | mg/kg |

2.2 Partial parameters assessment for contaminated sludges and sludge-soil mixtures

It is important to note that prior to phytoremediation, a partial characterization of the soil and sludge-soil mixtures was carried out. Furthermore, an assessment of the parameters for the sludge-soil mixtures was conducted after phytoremediation.

The pH determination procedure follows the methodology developed by Fernández et al., (2006). To conduct this procedure, 1g of dry and wet sludge is weighed separately in a 25 mL beaker. Then, 10 mL of distilled water is added to each beaker, stirred, and allowed to rest for 10 minutes. The pH meter is calibrated using buffer solutions, and the pH of the sludge samples is measured after 10 minutes. This analysis was performed in triplicate.

The electrical conductivity determination procedure is carried out according to the methodology developed by Fernández et al., (2006), and is described as follows:

- Preparation of saturation paste: 40g of dry and wet sludge were weighed in plastic containers, respectively. Distilled water was added with a burette and mixed with a spatula until saturation. The container was covered and left for three hours.
- Obtaining soil extract: The filter paper was placed on the funnel, moistened with distilled water, and the excess water was drained. The paste was then mixed again and placed in the funnel to be vacuumed.
- Measurement of electrical conductivity. The conductivity meter was calibrated with a standard solution before use. The electrical conductivity was measured, and three replicates were performed.

All procedures related to the determination of total petroleum hydrocarbons in dry and wet sludge are also carried out according to the methodology developed by Fernández et al., (2006). For this, a sample of 0.5 to 2g of sludge, previously crushed in a mortar, was weighed and placed in a 15 mL centrifuge tube to which 3g of anhydrous sodium sulfate was added. It was mixed vigorously in a vortex until homogenized (the anhydrous sodium sulfate must be dried in an oven at 120 °C for 4 hours). Then, 5 mL of dichloromethane was added and mixed again by vortexing for 45 seconds to ensure good solvent incorporation with the sludge. The mixture was centrifuged at 6,000 rpm for 10 minutes. The supernatant was removed and placed in a round-bottom flask. The sludge was washed two more times with the extracted solid residue until approximately 15 mL of supernatant (organic extract) was obtained. It should be noted that the container used should have a constant weight; for this purpose, the container was placed in an oven at 120 °C for 4 hours. It was removed and placed in a desiccator to cool. The container was then weighed, placed back in the oven, and the procedure was repeated until the weight did not change and the weight of the container was recorded (RA).

2.2.1. Quantification of total petroleum hydrocarbons

- Procedure: Once the organic extract obtained was in a round-bottom flask at constant weight, the total solvent (dichloromethane) was evaporated to dryness in a rotary evaporator at 740 ± 50 mbar and 45°C . The flask containing the solvent-free extract was reweighed, and the weight was recorded (RB). Hydrocarbon extraction and quantification procedures were performed in triplicate for wet and dry sludge samples.
- Calculations (TPH): The difference in weight corresponds to the total TPH content. To calculate the concentration of total petroleum hydrocarbons in the sample, the amount of soil weighed for extraction and the moisture content of the sample must be taken into account. The results should be calculated using equation 1, and expressed as milligrams of TPH per kilogram of dry soil.

$$\text{TPH (mg kg}^{-1}\text{ of d.s.)} = \frac{(RB - RA) * (CF)}{(P * FH)} \quad (1)$$

TPH (mg kg⁻¹ of dry soil) = total petroleum hydrocarbons in mg/kg of dry soil.

RA = weight (mg) of the empty container at constant weight.

RB = weight (mg) of the container with the concentrated organic extract.

P = amount of soil extracted (g).

FH = moisture correction factor (1 - (% moisture/100)).

CF = correction factor to convert to kg of dry soil = 1.000.

Calculations (% and removal rate)

The removal percentage corresponds to the amount of total petroleum hydrocarbons removed from the soil-sludge mixture divided by the initial amount of total petroleum hydrocarbons present in that mixture multiplied by 100. The results should be calculated using equation 2:

$$\text{removal percentage} = \frac{THi - THf}{THi} * 100 \quad (2)$$

THi = Total petroleum hydrocarbons present in the soil-sludge mixture at the beginning (mg/kg).

THf = Total petroleum hydrocarbons present in the soil-sludge mixture at the end (mg/kg).

In order to calculate the removal rate, it is necessary to consider the duration of the phytoremediation system implementation in hours and the total hydrocarbons removed during that time. This allows us to determine the amount of hydrocarbons removed per hour by applying a mathematical proportion (Fernández et al., 2006).

It is worth mentioning that the data analysis and statistical calculations in this study were conducted using the Microsoft Excel software program.

3. RESULTS AND DISCUSSION

In Table 2, the average pH obtained shows that wet sludge has a neutral pH, which is more favorable for the development of plants and microorganisms associated with the rhizosphere zone (Kim et al., 2006), while dry sludge has an alkaline pH and also higher

values of electrical conductivity, which hinders phytoremediation processes. The pH and electrical conductivity results decrease when mixed with soil.

Table 2. Initial pH and electrical conductivity of sludge and soil-sludge mixtures

| <i>Parameters (Unit)</i> | | <i>Values</i> | | | |
|------------------------------|-----------|-------------------|-------------------|--------------------------------|--------------------------------|
| | | <i>Dry sludge</i> | <i>Wet sludge</i> | <i>Dry sludge-soil mixture</i> | <i>Wet sludge-soil mixture</i> |
| pH (u) | R1 | 10.08 | 7.22 | 8.03 | 7.05 |
| | R2 | 10.04 | 7.25 | 8.12 | 7.16 |
| | R3 | 10.06 | 7.22 | 8.16 | 7.08 |
| | \bar{x} | 10.06 | 7.23 | 8.10 | 7.09 |
| | σ | 0.02 | 0.02 | 0.07 | 0.06 |
| | CV | 1.20 | 0.24 | 0.82 | 0.80 |
| CE ($\mu\text{S/cm}$) | R1 | 7462 | 440 | 7340 | 389 |
| | R2 | 7435 | 435 | 7305 | 375 |
| | R3 | 7453 | 445 | 7310 | 398 |
| | \bar{x} | 7450 | 440 | 7318 | 387 |
| | σ | 13.7 | 5.0 | 18.93 | 11.59 |
| | CV | 0.18 | 1.13 | 0.26 | 2.99 |

Note: R₁: initial result, R₂-R₃: replicas, \bar{X} : average, σ : standard deviation, CV: coefficient of variation

After phytoremediation (after 18 months), the values presented in Table 3 do not show significant variations compared to the results obtained before the phytoremediation system was established, except for the electrical conductivity in the mixtures of dry sludge and soil, where a reduction in values can be observed. This decrease can be attributed to the higher porosity and permeability of the dry sludge in comparison to wet sludge. The increased porosity of dry sludge facilitates the seepage and movement of water into the soil-sludge mixture (Giatman et al., 2019), promoting the absorption of contaminants and ions by plants and the leaching of ions from the system (Hacke et al., 2000). Consequently, the decrease in electrical conductivity occurs as a result of these processes.

The average pH of wet sludge remains more favorable for the development of plants and microorganisms associated with the rhizosphere zone, corresponding to neutral pH values.

Table 3. pH and electrical conductivity of soil-sludge mixtures after phytoremediation

| <i>Parameters (Units)</i> | | <i>Values</i> | |
|-------------------------------|-----------|--------------------------------|--------------------------------|
| | | <i>Dry sludge-soil mixture</i> | <i>Wet sludge-soil mixture</i> |
| pH (u) | R1 | 8.08 | 7.14 |
| | R2 | 8.15 | 7.20 |
| | R3 | 8.10 | 7.17 |
| | \bar{x} | 8.11 | 7.17 |
| | σ | 0.04 | 0.03 |
| | CV | 0.44 | 0.42 |

| | | | |
|----------------------------|-----------|-------|------|
| CE ($\mu\text{S/cm}$) | R1 | 304 | 383 |
| | R2 | 305 | 395 |
| | R3 | 279 | 376 |
| | \bar{x} | 296 | 385 |
| | σ | 14.73 | 9.60 |
| | CV | 4.98 | 2.49 |

Note: R₁: initial result, R₂-R₃: replicas, \bar{X} : average, σ : standard deviation, CV: coefficient of variation

The results of the determination of total petroleum hydrocarbons (TPH), in mg/kg, are shown in Table 4. It can be observed that the concentration of total petroleum hydrocarbons is higher in wet sludge (7%) than in dry sludge (4.3%). The concentration of these contaminants decreases when mixed with red ferralitic soil, although it still presents elevated values, which can interfere with the development and adaptation of plants. Hence, it is important to conduct preliminary studies to evaluate their adaptability to these adverse conditions.

Table 4. Total petroleum hydrocarbons in sludge and mixtures

| Parameter (Unit) | | Values | | | | | |
|---------------------|-----------|---------------|---------------|-----------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|
| | | Initially | | | | At 18 months | |
| | | Dry sludge | Wet sludge | Dry sludge- soil mixture | Wet sludge- soil mixture | Dry sludge- soil mixture | Wet sludge- soil mixture |
| TPH (mg/kg) | R1 | 43448 | 70405 | 14435 | 23412 | 5472 | 7452 |
| | R2 | 43387 | 70468 | 14492 | 23485 | 5498 | 7499 |
| | R3 | 43359 | 70334 | 14456 | 23492 | 5451 | 7457 |
| | \bar{x} | 43398 | 70402 | 14461 | 23463 | 5473 | 7469 |
| | σ | 45.51 | 67.04 | 28.83 | 44.30 | 23.82 | 26.06 |
| | CV | 0.10 | 0.09 | 0.20 | 0.19 | 0.44 | 0.35 |

Note: R₁: initial result, R₂-R₃: replicas, \bar{X} : average, σ : standard deviation, CV: coefficient of variation

After a year and a half of phytoremediation, we can see that the average total petroleum hydrocarbon levels are 5473 mg/kg in dry sludge-soil and 7469 mg/kg in wet sludge-soil, which are lower than the values obtained at the beginning of the planting stage, and this represents a 62% and 68% removal of hydrocarbons at a rate of $0.68 \text{ mg}\cdot(\text{kg}\cdot\text{h})^{-1}$ and $1.22 \text{ mg}\cdot(\text{kg}\cdot\text{h})^{-1}$, respectively. These values were expected to fall within a range exceeding 50% removal efficiency because they align with results from previous studies where different plants, such as rye (68%) and lolium (61%), were evaluated for phytoremediation of hydrocarbon-contaminated sludge (Muratova et al., 2008). The consistent removal rates achieved with these plants further support the viability of phytoremediation as a practical solution for addressing TPH contamination.

Although the TPH levels obtained decreased to less than half of the values obtained before implementing phytoremediation, these (5.47% by weight for dry sludge-soil mixtures and 7.47% by weight for wet sludge-soil mixtures) did not comply with NC 1263, (2018), which states that the hydrocarbon content must be less than 0.1% by

weight. The favorable results of the phytoremediation process emphasize its potential for long-term remediation efforts. By further optimizing plant selection, remediation techniques, and monitoring strategies, phytoremediation can be a valuable tool in mitigating the environmental impacts of hydrocarbon contamination and promoting ecosystem restoration.

Comprehensive assessment of the progress and adaptation of phytoremediation plants over two time periods:

To evaluate the progress of the phytoremediation plant, two time periods were taken into account. Firstly, after one month of planting to assess if the plant was able to survive high concentrations of hydrocarbons, and then after 18 months to examine its adaptation in terms of growth and reproduction in the environment.

When the survival of the *Cyperus alternifolius* plant was evaluated sensorially after 30 days, a slow growth dynamic was observed, with no noticeable changes in leaf thickness (Figure 2, B). During the first weeks, the plants lost their green color in the stems and showed a caramel coloration, with symptoms of not being able to withstand the contamination. However, after three weeks of planting, their recovery was observed as the first shoots began to sprout. After the first month of planting, these plants showed resistance to the presence of petroleum hydrocarbons in soil and drilling sludge mixtures at a ratio of 3:1.

After 18 months, the plant has shown a positive capacity for adaptation under the conditions of contamination to which it has been subjected to. Adequate growth in size and thickness of stems and leaves was observed, without showing signs of chlorosis or necrosis (Figure 2, C). The *Cyperus alternifolius* plant was able to reproduce in the environment.

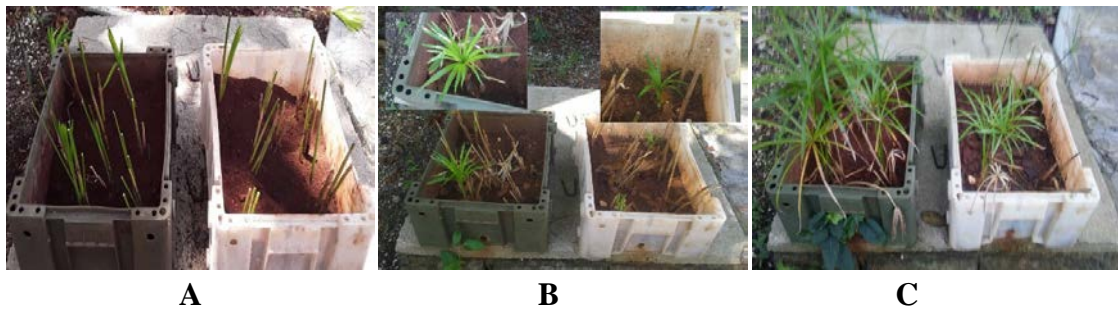


Figure 2. Experimental phytoremediation systems. Planting stage (A), after one month (B), and at 18 months (C). Source: Taken by the authors

4. CONCLUSIONS

1. The *Cyperus alternifolius* plant has shown resilience to petroleum hydrocarbons in both dry and wet sludge and has adapted well to adverse conditions. Despite initial challenges, the plant showed growth and recovery within three weeks and ultimately thrived for 18 months with no signs of damage. This resilience and successful reproduction highlight its potential in drilling sludge phytoremediation.
2. The *Cyperus alternifolius* plant effectively reduced total petroleum hydrocarbon levels by 62% in the dry sludge-soil mixture and 68% in the wet sludge-soil

mixture after 18 months of phytoremediation. While not meeting the regulatory standard, these results underscore the potential of phytoremediation for long-term remediation efforts. Further optimization will improve environmental impact mitigation and ecosystem restoration.

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CONFLICTS OF INTERES

The authors declare that there are no conflicts of interest.

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- PhD. Maira María Pérez Villar. Supervision, research, writing - review and editing.