Comparing the Electroremediation of Gleysol Soil Contaminated with Hydrocarbons with Triton X-114 Washing and Bioremediation with Solid Cultures Employing Agroindustrial Residues

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The continuously increasing population, in particular in large urban areas, and industrial and agricultural developments have caused direct and accidental soil contamination. In the last two decades, there have been significant advances in the regulation of soil pollution, parallel to the development of methodologies for evaluating and remediating contaminated soils. In Mexico, the most common remediation treatments for contaminated sites are biological, chemical and physicochemical. However, the appropriate remediation treatment for a site depends on the physicochemical properties of the contaminant and the soil. For this reason, Gleysol soil contaminated with hydrocarbons from the Nuevo Teapa, community in the southeast of the state of Veracruz in Mexico was characterized. After, this soil was used to test three different remediation treatments: soil washing with surfactant Triton X-114, biological treatment with solid culture and electroremediation with 0.1 M NaOH. Finally, the three treatments were compared in terms of removal efficiency, toxicology and cost.

1. INTRODUCTION

Soil contamination has increased in recent years due industrial growth, leading to contamination from the use of fertilizers and pesticides, metal deposition, and oil spills, as well as other human activity.

Nuevo Teapa is a community located to the north of the Isthmus of Tehuantepec, in the southeast of Veracruz in Mexico. This is an industrial area that is considered an important part of the

country, mainly due to oil (distribution, refinement, processing, etc.). Oil is basis of the economy and development there, which is the headquarters of the petrochemical industry, not only in Mexico but also in all of Latinoamerica. This area, due to industrial activities, has been involved in various environmental emergencies and widespread pollution [1]. The soil obtained from there, Gleysol has a swampy texture due to a considerable amount of fine material, which gives the material a "muddy" consistency that may mean that it becomes somewhat impermeable [2].

This has led to the implementation and dissemination of information related to its rehabilitation treatment, and to the creation of data sources listing treatment efficiencies and possible project costs [3]. There are three main types of treatments, thermal, biological and physicochemical, although in many cases, combining these processes improves remediation effectiveness. Soil remediation treatment techniques include electrokinetic remediation, soil washing and biological treatments using industrial waste (also called solid culture technologies).

Electrokinetic remediation is an emerging technology that takes advantage of the conductive properties of soil to separate and remove organic and inorganic pollutants in soil, sludge and sediment. It uses an electric field to remove charged species (ions) through the application of a low intensity direct current (on the order of mA cm^{-2}) between a negative electrode (cathode) and a positive electrode (anode), while generally saturating the matrix with water or another soluble salt that acts as an ionic medium [4 - 11].

The soil is washed when water is injected, with or without surfactants or extractants, into the soil to mobilize, extract and treat contaminants. This method takes into account the solubility of the contaminants in water; however, the removal of non-soluble compounds such as hydrocarbons requires the use of surfactants or extractants as mobilization agents [12]. Surfactants are able to improve the mass transfer of hydrophobic organic compounds from a solid phase to an aqueous one; they decrease surface tension and therefore improve the effectiveness of the washing treatment [6, 13 - 16].

Bioremediation has been preferably used for the degradation of organic compounds to dispose of organic wastes and produce enzymes, fungi, etc. [17]. Recent research reported satisfactory results with the use of solid culture technology, which uses composting strategy under a controlled process. Solid culture technology can treat soils and sediments contaminated with biodegradable organic compounds, and ultimately create stable innocuous byproducts [17 - 19].

By all the benefits of the three different methods mentioned before, this article compares electrokinetic remediation to soil washing and solid culture technology, measuring the removal rates of total petroleum hydrocarbon (TPH) and organic compounds before and after treatments. In addition, the three treatments were compared in terms of removal efficiency, toxicology and cost.

2. METHODOLOGY

2.1. Materials.

NaOH was acquired from Perkin Elmer, while Triton X-114, NH_4NO_3 and KH_2PO_4 were from Sigma. Bagasse and filter cake were obtained from a sugar factory for free. Analytical organic solvents (C_6H_{12} and CH_2Cl_2) were used for the Soxhlet techniques, GC-MS and Deltatox.

Soil samples were taken from a contaminated site in the community of Nuevo Teapa, Veracruz, at a depth of 50 cm, according to the procedures in the standard NMX2 - 12/1 - 1987b and to the procedure SW82 - EPA Chapter 9 while maintaining the temperature at 277 K. The sampling took place two years after that an environmental incident happened there, and had been treated previously [20].

2.2. Treatment of samples.

2.2.1. Electrokinetic remediation.

To carry out the treatment, an acrylic tubular reactor of 6 cm (length) by 3 cm (diameter) containing a 30 g sample of polluted soil (PS) was used. Once assembled, 0.1 M NaOH solution was passed through at a constant flow of 1.5 mL min^{-1} by using a peristaltic pump (Cole Parmer, Masterflex L / S Economy Drive, Model 7554-85).

To perform the electrokinetic remediation, a dimensionally stable electrode (DSA) using Ta_2O_5 | Ti as the anode (13.88 cm^2) and Ti mail (7.5 cm^2) as the cathode was used, with 6.5 cm between the electrodes. A 20 mA current was applied using a power supply (PC Power Supply, GP - 430BDO model) for 3.5 hours. During the treatment, the variation in the potential and pH was monitored.

2.2.2. Soil washing assisted by surfactant.

For the washing treatment, a similar reactor with the same sample weight of PS (30 g) was employed. A solution of 4 % v/v Triton X-114 was passed through the reactor at a constant flow rate of 1.5 mL min^{-1} by a peristaltic pump (Cole Parmer, Masterflex L / S Economy Drive, Model 7554-85) for 6 h [6].

2.2.3. Bioremediation.

Agroindustrial waste, such as bagasse and filter cake, was added to sterile serological bottles, while still maintaining the 100:2:2 soil-residue ratio. The adjusted nutrient ratio of C:N:P was 100:10:1, using ammonium nitrate (NH_4NO_3) and monobasic potassium phosphate (KH_2PO_4) as the nitrogen (N) and phosphorus (P) sources. Thirty grams of soil were deposited in vials and incubated at 301 K for 15 days, but were aerated every third day at a rate of 150 mL min^{-1} for 20 minutes to avoid anaerobiosis [20].

2.3. Characterization of samples.

The Soxhlet technique was used to extract TPH according to the procedure NMX - AA - 008 - SCFI - 2000. The semi-volatile organic compound determination was performed on a gas

chromatograph coupled to a mass spectrometer (Hewlett-Packard GC-MS) according to NMX - AA - 146 - SCFI - 2008.

To evaluate the toxicity of soil at the end of each of the three treatments, a Deltatox SDI Analyzer was used. For the analysis, 10 µL of each of the samples dissolved in hexane were taken, to which microorganism strains were added to measure effect. The tests were carried out under constant temperature and pH. In this study, a toxicity test was performed, which consisted of planting a bean seed (*Phaseolus vulgaris*) in 5 g of the 3 types of treated soil, adding 2 mL of water with sun exposure and observing the evolution and growth of the plant for 21 days.

3. DISCUSSION OF RESULTS.

Table 1 shows the removed compounds for each of the treatments when compared to the initial contaminated soil sample. Table 2 shows that electroremediation process removed the most hydrocarbon using 0.1 M NaOH as supporting electrolyte, having, at the end of the treatment, a soil TPH of 726 mg Kg^{-1}, equal to 81.9 % removal. Less efficient was soil washed with Triton X-114 surfactant, with only 11.9 % efficiency, leaving 3 522.8 mg Kg^{-1} of hydrocarbon in the soil due to the sorption of Triton X-114 into the soil matrix. In contrast, for the biological treatment, the removal percentage was 44.4 %, possibly due to the short period of time (15 days) in which the experiment was performed, as this method may require more time for better efficiency.

In the case of the presence of new compounds in the biorremediation process like the Benzoanthracene, Dotiacontane and Hexatriacontane with 0.24, 0.23 and 3.94 % respectively (Table 1) could be by the addition of the nutrients during the solid culture, but to be sure we need to make other analysis to probe it.

Table 1. Comparison of the remaining compounds found in the soil at the end of the treatments.

Compound	Area (%)			
	Polluted soil	Soil washing with surfactant Triton X-114	Bioremediation with solid culture	Electroremediation using 0.1 M NaOH
1-Octene	N.D.	N.D.	21.41	N.D.
1-Phenyl-1,3,3-trimethyl lindane	N.D.	N.D.	N.D.	0.33
Benzoanthracene	N.D.	N.D.	0.24	N.D.
Di-(2-ethylhexyl)phtalate	3.21	4.12	7.81	16.81
Dodecane	0.92	N.D.	N.D.	N.D.
Dotiacontane	N.D.	N.D.	0.23	N.D.
Fluorantene	2.13	0.14	0.51	N.D.
Hexatriacontane	N.D.	N.D.	3.94	N.D.
n-Eicosane	2.17	N.D.	8.27	1.69
n-Hexadecane	1.45	N.D.	1.01	N.D.
Octadecane	N.D.	N.D.	1.53	N.D.
Octylphenol etoxylate	N.D.	18.41	N.D.	N.D.
Pentatriacontane	N.D.	N.D.	0.73	N.D.
Phenanthrene	1.22	N.D.	N.D.	N.D.
Phyrene	2.17	N.D.	N.D.	N.D.
Tetrachloroetene	0.75	N.D.	N.D.	N.D.

N.D. = Not Detected.

Table 2. Comparison of the time, removal percentage, cost and toxicity after treating samples by bioremediation with solid culture, soil washing with surfactant Triton X-114 and electroremediation using 0.1 M NaOH.

Process	Time (h)	Remotion (%)	Cost ($ US dollars g soil^{-1})	Deltatox		Phytotoxicity
				%	Grade	Grown
Soil washing with surfactant Triton X-114.	5	11.9	4.48	86	High	Stem
Bioremediation with solid culture.	360	44.4	2.23	31	Low	N.S.
Electroremediation using 0.1 M NaOH	3.5	81.9	12.51	40	Moderate	Slave
Polluted soil.	4 200 mg Kg^{-1} HTP		-	90	High	-

N.S. = Not Shown.

According to NOM-138-SEMARNAT/SS-2003 (Mexican law), unlike for soil washing, both the biological and electrokinetic treatment results are under the maximum permissible limit for the heavy fraction in agricultural land use (3 000 mg Kg^{-1}). However, the sample also had to have a maximum permissible mean compound fraction of 1 200 mg Kg^{-1}, so the only treatment showed results under this limit was electroremediation (726 mg Kg^{-1}).

The surfactant is capable of solubilizing the hydrocarbon and removing it from the matrix, but also stays in the matrix after treatment. However, the soil to be treated, Gleysol, (which contains a substantial amount of clay as organic material) significantly decreases the efficiency of the wash, resulting in inadequate treatment for this soil type and therefore inefficient removal of hydrocarbons [21 - 23]. In this regard, Triton has surfactant-soil-contaminant interactions, undergoing sorption - desorption steps (solid - liquid interface). In this case, it only solubilizes the Fluoranthene compound, and transfers it to the liquid phase for removal and encapsulation in the formed micelles, transporting the pollutant out of the matrix for its removal.

Moreover, in the case of bioremediation, organic compounds may be degraded when consumed as a source of energy for the microorganisms and subsequently form novel chemical compounds (Table 1) that can be used by other microbe consortia for the same end. These new consortia can further degrade the starting compounds, until the substrate is "finished" or is a final compound that is non-biodegradable by the microorganisms [24 - 29]. At the end of the biological treatment, microbial consortia, such as those of *Aspergillus sp* and *Clostridium,* were observed, although their origin was not clearly understood, *i.e.* if they were native to the soil or were included because of the texturizers or additions such as bagasse and filter cake, which might be a possible biodegradation route.

Thus, the fungus (*A. sp.*) can incorporate Fluoranthene into its metabolic pathways through membrane oxidization and catalyzation by reactions with monooxygenase and epoxide hydrolase enzymes forming "trans" compounds for continuous Fluoranthene oxidation, and achieve degradation of the compound [27 - 28].

For electroremediation, the addition of 0.1 M NaOH is favored, primarily due to the increased medium conductivity, which stimulates electroosmotic flow, and secondly due to the increased solubility of TPH in basic medium, which improves mobility and contaminant removal [30 - 32].

TPH solubility in the presence of NaOH is from previous SOM attachments by weak bonds or Van der Waals forces, so that when the SOM is dissolved under basic conditions, the hydrocarbons are flushed out of the reactor (aided by electroosmotic flow, which has increased due to the Na^+ ions of the supporting electrolyte) and directed to the cathode where the highest concentration of hydrocarbon is [32].

Furthermore, hydrocarbons such as Fluoranthene are carried by the "solubility" part and surrounded by water molecules, for which electroosmotic flow and hydraulics are favored, possibly due to the presence of SOM solubilized by NaOH dragging them out of the reactor. Also, degradation of this compound near the anode could be possible, since this type of electrode can be employed as catalysts to oxidize organic compounds by $^\bullet OH$ radicals generation capable of breaking the double bonds of the molecule Phenanthrene [33].

According to Table 2, a greater hydrocarbon reduction was achieved using electroremediation treatment with 0.1 M NaOH. Reducing the concentration of n-Eicosane allows for the possible transformation to Di-(2-ethylhexyl)phthalate and possible generation of 1-Phenyl-1,3,3-trimethyl-lindane. The latter two compounds could also be generated by the electrochemical conversion of Tetrachloroethene, Dodecane, Phenanthrene, Fluoranthene, Pyrene and n-Hexadecane (Table 1).

Soil samples treated with each method were analyzed using ICP-MS to check for changes in element levels in the Gleysol soil after the processes (Table 3). Most of the elements slightly increased, although some differences could have been caused by addition of the elements as part of the treatments; for example, an increase in K^+ in the biological treatment could be from adding nutrients and Na^+ in the electrokinetic treatment from adding NaOH. Despite no major changes to the elemental composition of soils treated with different methods, we can infer that the manipulation before and during treatment as well as the processes themselves allow the elements that were blocked by the contaminant to be spread, either by microorganisms or electrokinetic phenomena.

Table 3. List of the chemical elements present in the soil at the end of the treatments.

Element	Concentration (mg Kg^{-1})		
	Soil washing with surfactant Triton X-114	Bioremediation with solid culture	Electroremediation using 0.1 M NaOH
As^{3+}	1.02	1.32	1.09
Ca^{2+}	4 892.00	26 949.34	26 078.46
Fe^{2+}	17 532.89	18 167.68	17 115.37
K^+	1 285.19	4 426.27	969.31
Mg^{2+}	2 535.34	2 869.34	1 968.88
Mn^{2+}	168.75	193.02	232.28
Na^+	811.01	845.51	2 488.62
Ni^{2+}	3.79	325.60	8.69
Ti^{2+}	100.37	88.46	86.85
Tl^{2+}	10.99	10.99	10.99
V^{2+}	40.18	33.98	31.18

Since the treatment that showed better HPA removal efficiency was electroremediation with 0.1 M NaOH (81.9 %), changes in soil due to the treatment were characterized. The parameters or characteristics tested were: texture, pH, organic matter, conductivity and cation exchange capacity. The sandy loam soil texture of uncontaminated soil was modified by the presence of hydrocarbons and became characterized as clay loam. This texture did not change due to electroremediation.

As for pH values, uncontaminated soil had an acidic pH (5.21), which was neutralized by the presence of hydrocarbons (7.21), and subsequently basified (9.02) during electroremediation from the addition of the 0.1 M NaOH supporting electrolyte.

However, despite the impact this may have caused the soil under study, the same electroremediation system could neutralize treated soil back to acceptable pH levels according to an existing pedological concept, because in the same manner, the hydrolysis of water would generate new pH gradients to neutralize it.

For SOM, no significant differences were observed between clean and electroremediated soil, which eliminates the possibility that during the electrochemical treatment, SOM is removed together with the contaminants. This result is important, since we found that soil electroremediation occurred between the pores of the soil by electromigration and electroosmostic processes without affecting the surface of the particles where the SOM is found.

When hydrocarbons were present in the contaminated soil, conductivity values decreased considerably due to the hydrocarbons surrounding soil particles and prevented the release of contained ions between each particle. After electroremediation, the soil conductivity increased from 1×10^{-5} dS m^{-1} to 0.56 dS m^{-1}, *i.e.* approximately five times the value obtained from clean soil (0.101 dS m^{-1}) due to the addition of NaOH to the system.

The CEC showed twice the amount of hydrocarbon present in contaminated soil (21.21 Cmol Kg^{-1}) compared to clean soil (10.21 Cmol Kg^{-1}) due to an aggregation of soil particles with HPA, since upon making the determination and breaking these aggregates, occluded cations are released into these aggregates.

However, after the electroremediation of contaminated soil, CEC slightly increased (24.53 Cmol Kg^{-1}) due to the addition of Na^+ from NaOH, suggesting that both structurally and in cation content, the application of the electric field during electrochemical treatment did not significantly affect chemical composition.

Using NOM-021-RECNAT-2000 to evaluate soil health or fertility involved measuring several factors such as pH, SOM content, CEC, inorganic nitrogen, phosphorus and micronutrients (Fe, Mn, Zn) as well as metal contaminants (such as Pb, Cd, Ni). The results for electroremediated soil fall within the acceptable mean values of SOM, CEC and low salinity, despite increasing the conductivity of the soil when compared to pretreatment levels. ICP-MS showed that the amount of Ni is low while the amount of Fe is significant.

At the end of treatment, a high alkalinity (pH = 9) was recorded, which could affect the soil. However, the study zone has high levels of rainfall which acidifies the soil naturally, such as with SL, and hydrolysis can be induced to reduce or neutralize soil pH, to create treated soil that is acceptable for use.

Moreover, Table 2 shows the results for each of the samples and their level of toxicity according to the equipment manual. Note that the determinations were performed in duplicate. When tested, uncontaminated soil had a toxicity of 10 %, below the percentage classification limit of low toxicity. According to the equipment manual, 49 % to 70 % is moderately toxic and above 70 % is highly toxic. Contaminated soil showed 90 % toxicity, confirming the need for further treatment.

Furthermore, evaluating the toxicity of the extracts obtained from each of the treated soils shows that the soil from the biological treatment had a low toxicity (31 %), the soil from the electrochemical treatment a moderate toxicity (40 %) and the soil from washing a high toxicity (86 %) due to the surfactant that remained in the matrix.

Biological treatment resulted in lower toxicity than the other treatments, suggesting that the initial compounds were degraded to less toxic compounds (results which follow the principles of green technology) despite generating more compounds from a partial degradation of the initial compounds [17 – 18, 20, 24, 34 - 37]. While the soil obtained after the electroremediation treatment is considered moderately toxic, the remaining compounds are the same as those found in the contaminated soil but present in smaller amounts.

Moreover, in addition to the above analysis, another toxicity test was performed by planting the bean seed *Phaseolus vulgaris* in 5 g of soil treated by each the three treatments over a period of 21 days. Two mL of water were added daily for growth and the plants were exposed to the sun.

Figure 1 shows the growth of the bean seed for each of the samples. Maximum growth is observed on day 14 for the soil sample treated with 0.1 M NaOH electroremediation. Growth was less for the plant grown in the soil washed with Triton X-114 surfactant, and plant color was darker. However, the seed ´planted in soil that had been biologically treated with solid cultures did not grow at all, possibly due to competition between the adapted microorganisms and bean seed for nutrients. Table 4 compares the sizes of each part of the bean plant (root, stem and leaf) at the end of the experiment, verifying greater growth for plants grown in the electrorremediated soil.
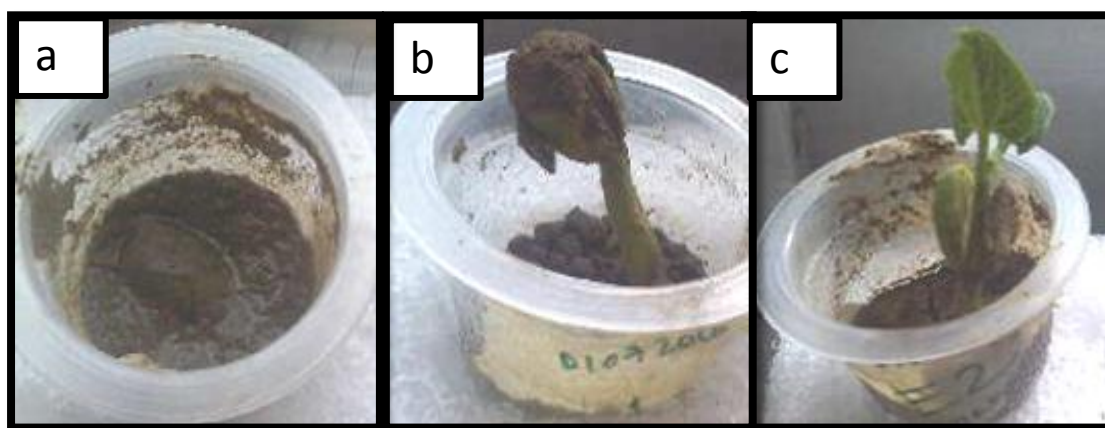

Figure 1. Photos of bean seed growth in the samples treated by: (a) bioremediation with solid culture, (b) soil washing with surfactant Triton X-114 and (c) electroremediation using 0.1 M NaOH.

Table 4. Comparison of the root, stem and leaf sizes for bean plants grown for 21 days in the treated soils.

Treated Soil	Plant Part (cm)				
	Root	Stem		Leaf	
	Size	Thickness	Height	Width	Height
Soil washing with surfactant Triton X-114	1.2	0.2	2.1	N.S	N.S.
Bioremediation with solid culture	N.S.	N.S.	N.S.	N.S.	N.S.
Electroremediation using 0.1 M NaOH	0.8	0.2	4.0	0.7	1.0

N.S. = Not Shown

Electroremediation improved edaphological soil properties due to electrokinetic phenomena which helped disperse the micronutrients that the bean seed species *Phaseolus vulgaris* requires for proper growth and development. When compared to soil washing and solid cultivation, these results also disprove that the treatment sterilized the soil.

In addition to comparing the three treatments by their hydrocarbon removal efficiency for Gleysol soil, an economic study was conducted at the laboratory to measure the expenditure incurred for each during the experiments, taking into account all the factors involved with the treatments. The overall final cost of each treatment for a sample of 30 g of soil, which was the quantity used for the experiments is shown in Table 2, along with the time, percentage of removal, and toxicity recorded for each of the treatments during the study.

Biological treatment with solid culture cost only $ 2.23 US dollars for the remediation of a soil contaminated with hydrocarbons and had low toxicity (31 %). However, the removal took a very long time (360 h) for only 44.4 % removal percentage. While soil washing with surfactant had an intermediate cost of $ 4.88 US dollars, and needed only a short time (5 h) to achieve 11.9 % hydrocarbon removal efficiency, it resulted in high toxicity (86 %), due to the surfactant that remains in the soil matrix after the treatment.

On other hand, the electroremediation treatment was the most expensive at $ 12.51 US dollars, this method needed less time (3.5 h) to achieve higher hydrocarbon removal (81.9 %) with only moderate toxicity (40 %). The majority of the increased cost was from the materials used in the process, materials which also have lifetimes greater than 5 years.

4. CONCLUSIONS.

According to the results of the Gleysol soil characterization, we can infer that the presence of hydrocarbon may intervene in the measurement of various physical and chemical parameters. Such is the case with the texture, where the adsorbed hydrocarbon in the matrix and largely organic matter

form small aggregates with small particles, which have a larger sorption area which creates an apparent density, and slows the sedimentation rate.

Likewise, the pH value is modified by the presence of hydrocarbons which can obstruct the exchange and passage of protons and ions generally present in soil particles, which in times of runoff are "washed" into groundwater. Furthermore, parameters such as electrical conductivity and cation exchange capacity are affected by the presence of hydrocarbons in the soil particles, limiting the ability to measure the suspension of soluble ions.

As for the different treatments tested for the removal of hydrocarbon contaminated soil:

- For soil washing with the Triton X-114 surfactant, the amount of SOM, clay particles and fine particles found in Gleysol soil limited contaminant removal and created new contamination from the sorption of the surfactant. While the toxicity of the soil did not inhibit growth of a bean seed *Phaseolus vulgaris*, surfactant traces that remained in soil increased the toxicity. In addition, this treatment was moderately expensive compared to the other two treatments.

- For biological treatment with solid cultures using bagasse and filter cake as texturizing agents and a means of adaptation for the microorganisms *Aspergillus sp.* and *Clostridium*, a hydrocarbon removal percentage of 44.43 % over a period of 15 days was observed. Although the effectiveness of this treatment was limited by the conditioning time for microorganisms and the overall treatment time, the conditions were adequate for the process, which proved to be more efficient than the soil washing. However, the degradation of the hydrocarbons generated many intermediates during the process, which remained in the soil at the end of treatment (but showed no toxicity). While this treatment method was the cheapest of the three methods, *Phaseolus vulgaris* did not grow in these conditions.

- Electroremediation treatment using 20 mA, a flow of 1.5 mL min^{-1} of 0.1 M NaOH and IrO$_2$ - Ta$_2$O$_5$ | Ti as the anode and Ti mail as the cathode achieved better hydrocarbon removal efficiency (81.9 %) than soil washing with Triton X-114 (11.93 %) and bioremediation with solid culture (44.43 %). The electrochemical method was improved by adding a 0.1 M NaOH electrolyte, which increased the removal efficiency by increasing the conductivity of the medium so that it favored electroosmotic flow, which allowed improved hydrocarbon removal. NaOH solubilized the organic matter, and after the hydrocarbon adsorption, the contaminant was moved by hydraulic and electroosmotic flow, for an 81.9 % removal rate after 3.5 hours. Although electroremediation was the most expensive method of the three, this treatment resulted in fewer by-products compared to the other two, and showed only moderate toxicity, which did not affect soil properties nor inhibit the growth of the *Phaseolus vulgaris* bean plant.

For the remediation of Gleysol soil contaminated with hydrocarbon from Nuevo Teapa, located in the southeast of Veracruz, electroremediation was the most expensive method, but showed only moderate toxicity and achieved results in less time than soil washing with Triton X-114 and bioremediation with solid culture using agroindustrial wastes.

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