Flotation kinetic tracking of sand contaminated with hydrocarbons: image analysis of pulp color changes in time

Claudio Acuña [1], José Humire [2], Claudio Leiva [2,3]

[1] Department of Chemical and Environmental Engineering, Universidad Técnica Federico Santa María, 2390123, Valparaíso, Chile

[2] Departamento de Ingeniería Química y de Medio Ambiente Universidad Católica del Norte, 1270709, Antofagasta, Chile

[3] Oulu Mining School, University of Oulu, 90570, Oulu, Finland

Corresponding author: cleiva01@ucn.cl (C. Leiva)

Abstract: Hydrocarbon contamination in the environment poses a significant challenge, and various control methods have been explored. Soil remediation by flotation has been proposed as an effective approach. This method involves the separation of hydrophobic compounds, such as soil hydrocarbons, by introducing air into a stirred reactor containing the soil pulp designated for remediation. Experiments were conducted using a 5 L Batch flotation cell to evaluate operating conditions. These experiments focused on obtaining flotation kinetics with different organic mixtures, including fine sands measuring under 150 μm. The experimental design encompassed airflow, hydrocarbon concentration, and surfactant dosage. The research utilized a diluted pulp (3% solids) with a high organic concentration (8 and 17 g/L). Flotation kinetics were measured by developing an innovative technique based on pulp color and image processing software. This technique facilitated the tracking of concentration changes over time under Beer-Lambert's Law. Subsequently, the results were adjusted using kinetic models commonly employed in mineral flotation, including the first-order, Kelsall, and Klimpel models. This comprehensive analysis sought to elucidate the underlying phenomenology and assess the potential for industrial-scale implementation. The laboratory findings indicate the possibility of achieving recoveries of up to 87%, with a first-order kinetic constant of 0.7 (1/min). Both the gas flow rate and the addition of surfactant exert substantial influence on this constant, consistent with the observed phenomenology of this study. This study explores the integration of image analysis in flotation for hydrocarbon-contaminated soil remediation. The research aims to optimize remediation strategies by examining variables such as Beer-Lambert's law and prevalent kinetic models. The study focuses on scalable, eco-friendly decontamination methods, emphasizing enhanced process control and comprehension within soil flotation systems.

Keywords: flotation, organic, image analysis

1. Introduction

Hydrocarbons correspond to a main force of energy at this present time: For this reason, the environmental pollution caused by petroleum and petrochemical products (complex mixtures) is recognized as one of the most critical problems (Diaz et al.., 2001; Lee, 2003; Maltoni, 1997; Mohamed, 2007; Moosai and Dawe, 2003; Raiger and Lopez, 2009; Schmidt, 2001), especially when it is associated with large-scale accidental spills (Plohl and Leskovsek, 2002), as well as other industrial activities in which this element is required.

Once this pollutant has been spilled on the ground, most of the aphylactic compounds are lost by volatilization while some hydrocarbons, such as polycyclic, persist on the surface, (Jimenez et al.l, 2010) producing a significant impact due its toxicity and recalcitrant effects on living beings (Tissot and Welte, 1984). The damage from hydrocarbon pollution depends on the type and concentration of the pollutant and these discharges pose direct significant risks to human health and environment. Furthermore, it undermines the security of livelihoods in the long term (Baumuller et al., 2011).

For the pollution to be remediated exists several techniques and/or methodologies, which, in first place, they can be classified as in situ and ex situ and, there are three treatment categories in which those of physical-chemical, biological and thermal nature are distinguished [Pandey et al., 2009; Seoanez, 2000; Sutton et al., 2011; Urum et al., 2005; Velasco and Volke, 2003).

Among the treatment alternatives, there is the flotation process, a method widely applied in mining used for different compounds removals due to its versatile extraction characteristic, applied in a wide field (Rubio et al., 2001), as in soils, sediments, residues and sludge cases.

In the case of contaminated soils with hydrocarbons, it has been demonstrated that dissolved air flotation is effective in the removal of these components (Zhang et al., 2001; Mouton et al., 2008), in which it is required a specific control of variables that affect the process to optimize their efficiency (Acuña, 2007; Grau and Heiskanen, 2002; Gorain et al., 1998; Grau and Heiskanen, 2003). In order to help the variable control and optimize the floating system, an image analysis system is tested, in which, through colour changes plus contrasts on the pulp, it was possible to measure concentration throughout the time based on Beer-Lambert's law. The results were adjusted based on kinetic models frequently used in mineral flotation (first order, Kelsall and Klimpel), used to analyze the phenomenology and perform the scaling of the industrial application.

In this manner, flotation process characteristics, such as the use of reduced amounts of reagents, low energy requirement, and finally, a reduced application time (Navarra et al., 2009; Finch and Dobby, 1991; Finch, 1995; Mendez, 2009; Gorain et al., 1997; Nesset et al., 2005), indicate a broad control of most of their variables. These are aspects that in organic flotation are also desired to obtain; thus, they can serve as a comparison with the described remediation techniques.

This study explores the integration of image analysis in flotation for hydrocarbon-contaminated soil remediation. By scrutinizing variables via Beer-Lambert's law and prevalent kinetic models, it aims to optimize remediation strategies. Leveraging flotation's efficacy—minimal reagents, low energy demand, and swift application—the focus centers on scalable, eco-friendly decontamination. Enhanced process control and comprehension within soil flotation systems are pivotal. Through this exploration, the study seeks to refine remediation approaches, offering a pathway towards sustainable, efficient, and industrially applicable solutions for soil decontamination, aligning with the complexities of flotation dynamics in hydrocarbon removal.

2.   Materials and methodology

2.1. Equipment and materials description

Experiments were conducted using a 5 L Batch flotation cell to evaluate operating conditions. These experiments focused on obtaining flotation kinetics with different organic mixtures, including fine sands measuring under 150 µm. The experimental design encompassed airflow, hydrocarbon concentration, and surfactant dosage. The research utilized a diluted pulp (3% solids) with a high organic concentration (8 and 17 g/L).

In Fig. 1, a global instrumentation scheme and materials used for flotation tests is presented:
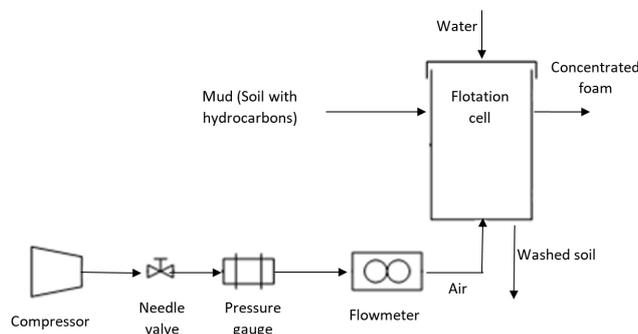

Fig. 1. Instrumentation scheme for pilot scale flotation tests

2.1.1. Laboratory flotation machine and accessories

The laboratory flotation machine was operated for 12 minutes per experiment at a stirring speed of 1000 rpm. The air was injected from a compressor and passed through the instrumentation, needle valve,

manometer, and flowmeter before being injected into the flotation cell. The needle valve was regulated to control a low and stable amount of air, and the manometer was set for a pressure of 3 Mpa. The air flow supplied was 3 and 6 (L/min), which was indicated by the flowmeter, and this was useful to define airflow in superficial gas velocity (cm/s). The flotation tests were conducted in a transparent acrylic cell with a 5 L capacity (Fig. 2), and a total volume of 4.5 liters was placed in the cell to place the contaminated soil with water and the reagent. The contaminated soil sample was made from two components: 150 gr of sand-type quartz mixed with bunker oil was used (at 40 gr and 80 gr levels), which was previously dissolved in commercial diesel at 10 %p/p. The amount of reagent was defined for two cases, low and high concentrations. For the first case, 50 mL of polyglycol frother (X133) a, 50 ppm, and 50 mL of commercial detergent diluted in water at 10% w/w were used. For the second case, the same amount of frother, 50 ml, and 100 ml of commercial detergent were used with the same concentrations.
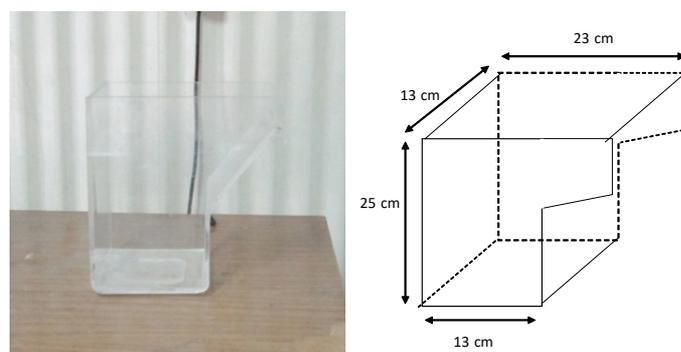

Fig. 2. Flotation cell dimensions

2.2. Experimental set-up

Firstly, the contaminated soil, water and reagents are placed in the cell; subsequently, the cell is located at the base of the flotation machine arranged for that purpose.

Later on, the mechanical stirrer of the flotation machine is lowered; thus, it remains completely submerged into the cell. At this point, the flotation machine's engine is turned on at a stirring speed of 1000 rpm and, it is kept on for 1 minute; then, the motor is stopped. The reagents are conditioned for 3 minutes with the contaminated soil. At the end of this period, the kinetic flotation test has started for a time of 12 minutes. The stirring speed of 1000 rpm is applied, and air is injected at a flow of 5-10 liters per minute. For each 12-minute experience, a video recording, focused on the cell, is made, showing the degradation of hue due to the decrease in concentration. Thereupon, the recordings were used for image conversion and image processing. The following Fig. 3 shows the material balance that represents the experience carried out.

2.3. Experimental design

Eight kinetic flotation tests were performed, which were developed based on an experimental design summarized in Table 1.

Table 1. Experimental design of kinetic flotation tests

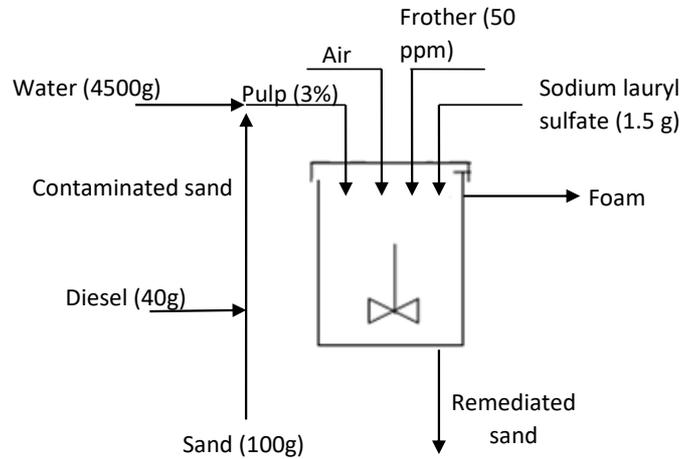| Concentration diesel (g/L)	Sodium lauryl sulfate concentration (g/L)	Aeration (L/min)
8.8	0.3	3
		6
	0.7	3
		6
17.6	0.3	3
		6
	0.7	3
		6

Fig. 3. Conceptual material balance of flotation test N°1

The applied operating parameters are described in Table 2. These parameters were kept constant for all the 8 experiences during the total time of application of the kinetic tests.

Table 2. Operation conditions for flotation tests

Fixed operation parameters	Value
Operation time (min)	12
Stirring speed (rpm)	1000
Water (g)	3500
Cell Volume (L)	4.5
Cell volume capacity (L)	5
Fine sand (g)	100
Fine sand size (mm)	0.5 – 1.5
Polyglycol X-133 (mL) diluted at 50ppm	50

2.4. Methodology development for kinetic measurement by light intensity variation

Fig. 4 shows a diagram of the methodology used to collect information from the flotation tests performed, in which a video recording of the flotation cell in operation with the flotation machine was made. For this, the light was placed on one side of the cell, and on the other, the brightness was captured with the video camera. The technique is based on the changes in image light intensity. As bubbles collect dark particles (hydrocarbons), the pulp color decreases in intensity. Given the background illumination, these variations can be directly measured as increased light intensity. Applying Beer-Lambert's law relates the concentration of color molecules with the absorbance, which is the logarithm of transmittance.

Subsequently, the collected video recordings were processed and converted from video to images, in which an image was obtained for time intervals of 5 seconds (Fig. 5). The time frame for data acquisition in a spectrophotometric analysis can be adjusted according to the kinetics of the process,
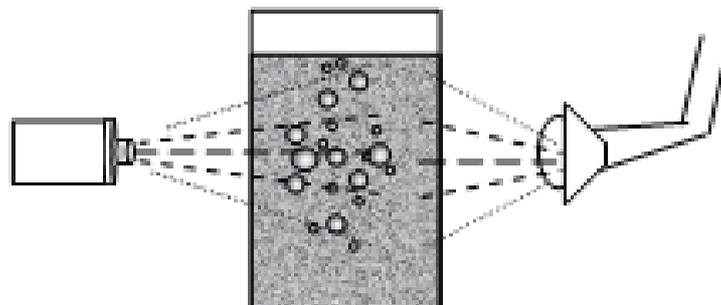

Fig. 4. Brightness captured through a video camera

with a maximum rate of 10 frames per second for a standard video camera. However, the time frame selection is based on the minimum detectable change in intensity, with a spectral resolution of 8 bits for each color band (red, green, blue) and 255 intensity levels.

The described method for analyzing the concentration of diesel in a sample is based on luminosity estimations using the Beer-Lambert law. The law quantifies the absorbed radiation as a function of molecules of the components or the element of analytical interest in a sample. In this case, the component of interest is the concentration of diesel. The method involves analyzing images obtained from the sample, where a determined luminosity is observed and subsequently analyzed by software to determine its specific intensity, given a background illumination, which, without dark elements, it has 100% transmittance. The results of the images obtained are shown in Fig. 5, where the luminosity increase can be observed over 200 seconds. Fig. 5 presents images from left to right, continuing from the first to the last row. The Beer-Lambert law is based on the relationship between the transmittance, which is the light's intensity being measured, and the body that is contaminated relative to the intensity of a target. In the absence of the contaminant, the logarithm of that results in the absorbance, which sustains a linear relation with the concentration of the organic matter. The image in the analysis comprises three color bands (red, green, and blue), each with 8 bits of spectral resolution and 255 intensity levels. For this specific application, the red band was utilized.
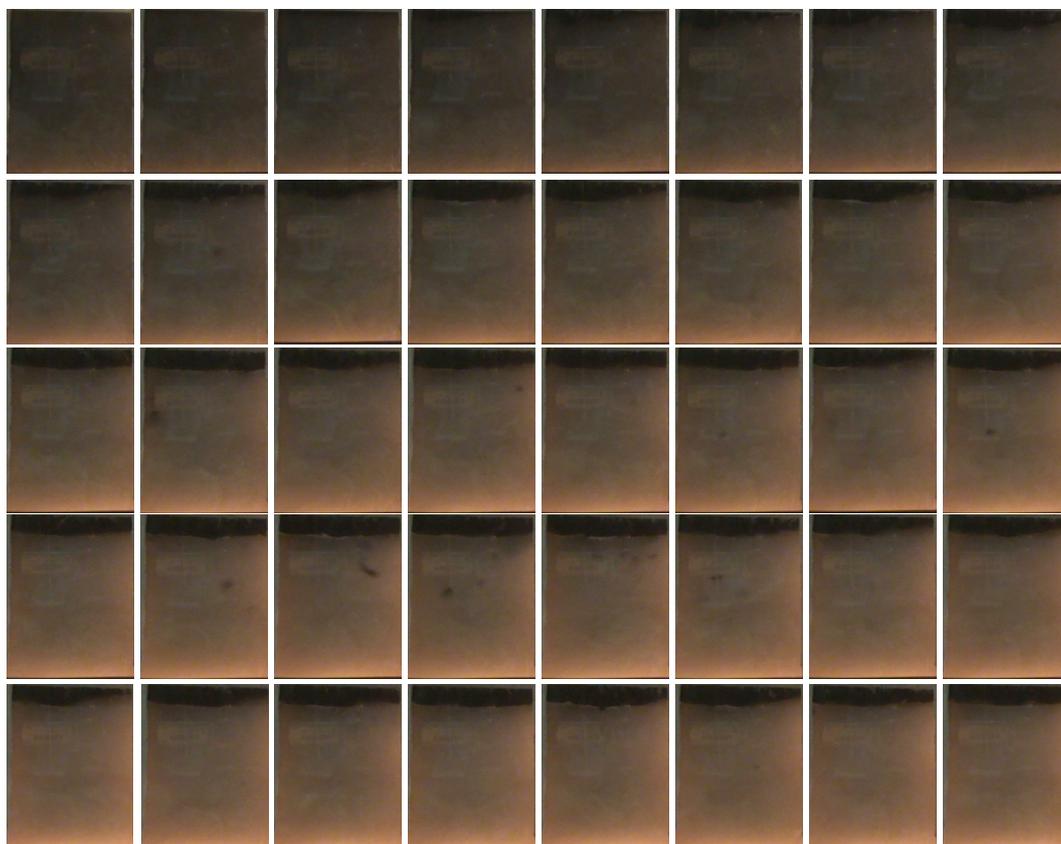

Fig. 5. Processed photographs

The equation representing this luminosity phenomenon is described below - Beer-Lambert law (Skoog and Leary, 1994):

$$A = +Log\left(\frac{I_o}{I}\right) \tag{1}$$

where: A – absorbance, I_o - beam radiation intensity in photons, which influences the cell, I - beam radiation intensity, which leaves the cell

$$A = \varepsilon\,C\,l \tag{2}$$

where: A – absorbance, ε - molar absorption coefficient, C - molar concentration, l - optical path length.

From the intensity values obtained and, with the described equation, which includes intensity, it is possible to determine the absorbance. Fig. 6 and 7 shows graphics of intensity and absorbance with its

corresponding time. From the previous graph, it must be considered that the intensity value was also obtained by operating the process only with water and sand, which generated a value of 240 as a result of intensity.

Subsequently, from the absorbance obtained, the transmittance can be determined using eq. 3. The graphical result of the transmittance with its corresponding time is presented in Fig. 8.

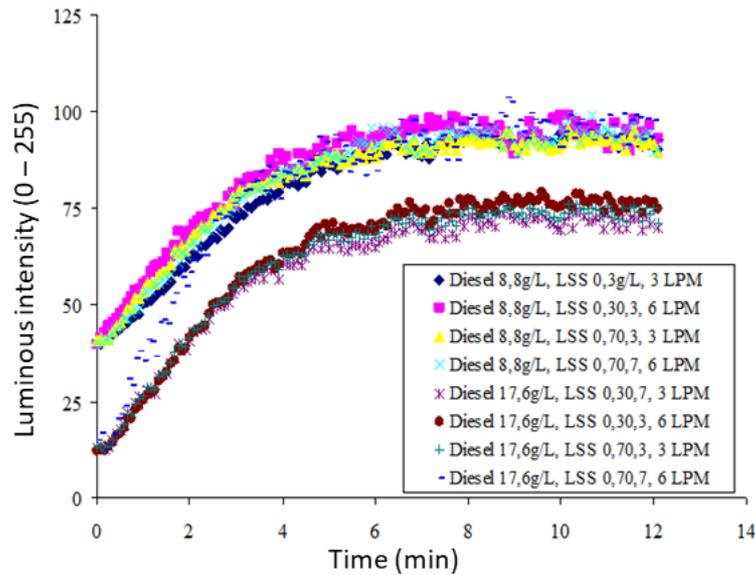$$A = -Log(T) \tag{3}$$

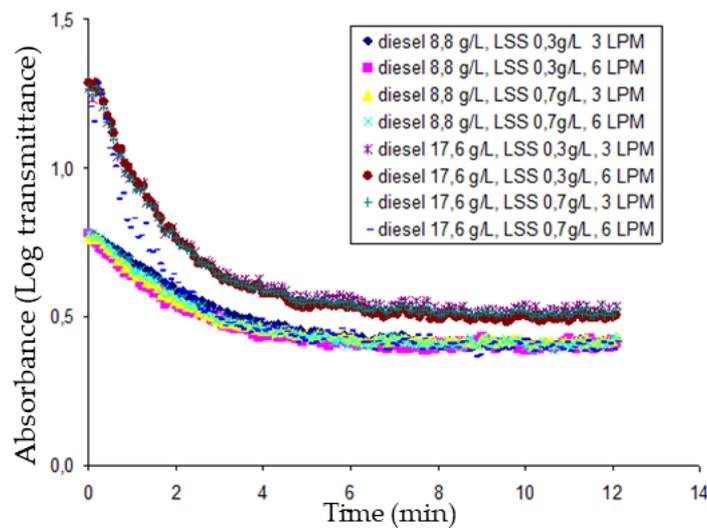Fig. 6. Intensity versus time

Fig. 7. Absorbance versus time

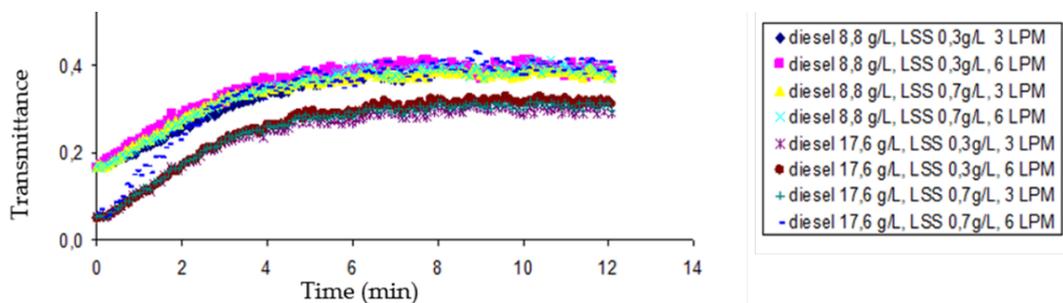Fig. 8. Transmittance versus time

Finally, by means of equation 4, it is possible to obtain the concentration (on a logarithmic scale)

$$C = m\, Log(T) + b \tag{4}$$

where: C - molar concentration, m – slope, T – transmittance, b – intercept.

3. Results and discussions

The flotation tests were processed, converting results from light intensity to absorbance, transmittance and finally, concentration and recovery of diesel. From this procedure, the kinetic curves for the 8 tests were obtained.

3.1. Kinetic flotation results

The graphics obtained for the 8 flotation tests are presented in Fig. 9.
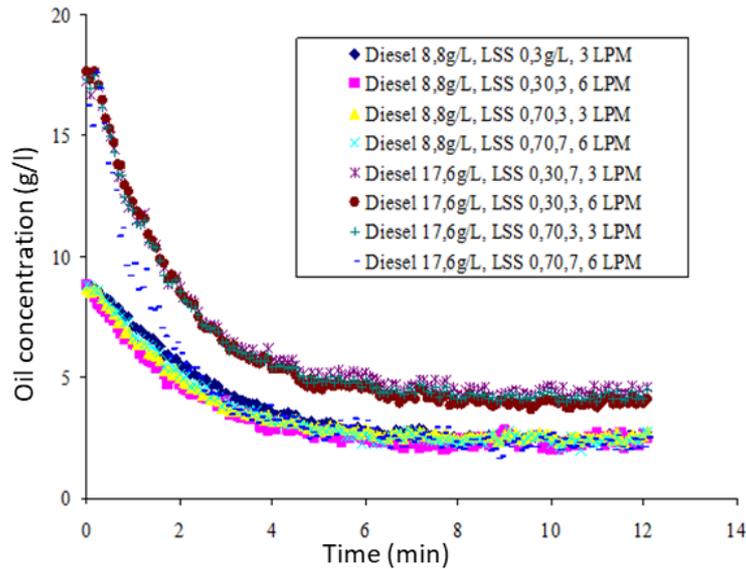

Fig. 9. Plotting of kinetic curves in flotation tests

In the graphic of Fig. 9, it is shown the 8 curves with the operating conditions applied and it can also be observed two main groups of curves that start at different concentration values (8,8 and 17,6), which indicates the initial difference. From the graphic of Fig. 10, it can be observed, in general, that all the curves reach a near-maximum recovery value. Nevertheless, in their initial section, some curves and/or flotation tests reach their maximum recovery in less time.
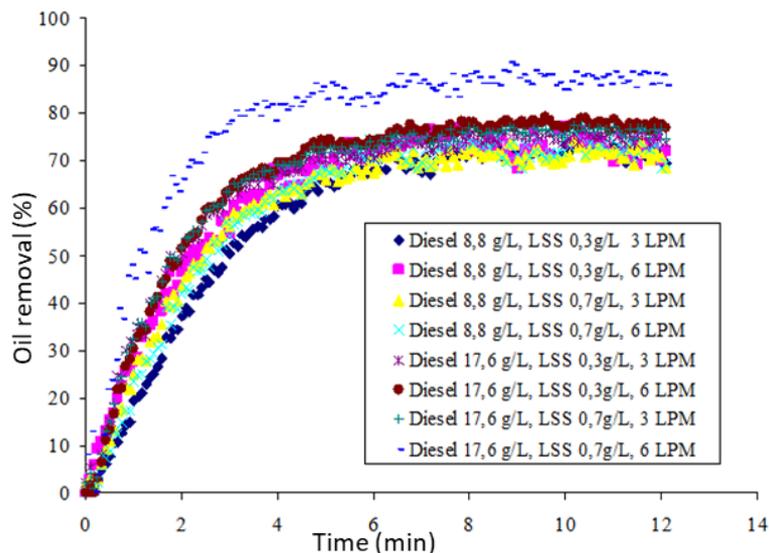

Fig. 10. Organic removal versus time

From the graphic, in synthesis it can be derived that with the exception of a single curve, all the rest reached over 75% of recovery within a time of 7 minutes.

Afterwards, with the results of the experimental curves, an adjustment of kinetic parameters was made for the three models with the 8 flotation tests performed.

3.2. Kinetic parameters adjustment

The adjustment of parameters for the case of first order models was performed for the kinetic constant value and and/or infinity recovery. For Kelsall model, the parameters were the value of fi. Finally, for Klimpel model, the adjustment was made for kinetic constant value (uniform distribution) and infinite recovery.

The adjustment results were obtained for the three models. Each flotation test performed (out of a total of 8) delivered an adjustment. In this way, a total of 24 adjustment results were obtained.

From lower squared error value, it was determined that first order model present the best fit (according to their lower squared error value of 588). For Kelsall model, the value of Kf was near to zero, in this manner, it was equivalent to first order model. Finally, Klimpel model had a value of 1569.

3.3. Experimental and adjusted curves.

Furthermore, the graphs were obtained where it can be observed that adjustment differences between the kinetic models with respect to the experimental curves are not noticeable. Fig. 11 indicates the result for kinetic test number 1.
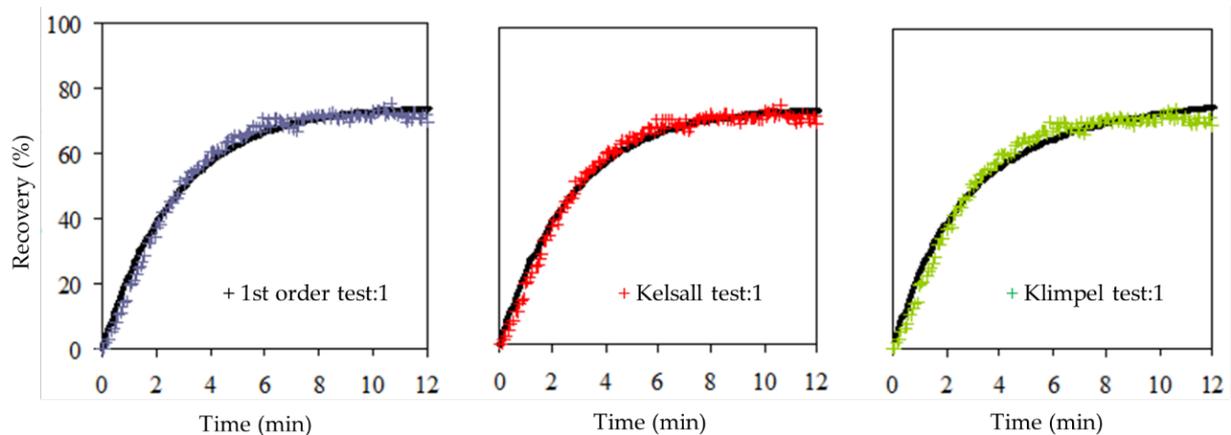

Fig. 11. Plotting of kinetic curves in flotation tests

3.4. Kinetic models

With the parameter adjustment obtained, throughout the minimization method by iteration of the sum of the squares of the residuals, it was determined that kinetic model (1° Order, Kelsall and Klimpel), for the 8 flotation tests carried out. With the calculated kinetic parameters, the following kinetic models are obtained (Eq. 5, 6 y 7).

$$R = 74.7\left[1 - e^{(-0.362t)}\right] \qquad \text{First order} \tag{5}$$

$$R = 74.7\left[1 - e^{(-0.362t)}\right] \qquad \text{Kelsall} \tag{6}$$

$$R = 74.7\left[1 - \frac{1}{1.013t}\left(1 - e^{(-1.013t)}\right)\right] \quad \text{Klimpel} \tag{7}$$

3.5. Effects of the applied variables

A covariance analysis was performed according to the following factors: air flow, sodium lauryl sulfate concentration (SLS) and diesel concentration. As a response to variables, the values of kinetic constant and maximum recovery were considered.

The Kelsall model, because of the adjustment, approached the first order model, thus, the analysis was established only for first order and Klimpel models. Table 3 indicates the summary with the values standardized to a value of 100.

Table 3. Standardized correlation for first order and Klimpel models

Model	Kinetics parameter	Air flow	Sodium lauryl sulfate	Diesel concentration
1º Order	K	0.362	0.377	1
	R_∞	74.7	34.6	82.6
Klimpel	K_m	1.013	1.055	2.977
	R_∞	74.7	34.6	82.6

From Table 3, the correlation between the response variable and factors, the following is inferred. For the first order model, the diesel concentration factor was the most influential for the kinetic constant value. Regarding the maximum recovery value, the interaction in diesel concentration and aeration factors obtained the most significant correlation and/or most influential factor. Fig. 12 shows the correlation obtained for the first order model, for the interaction of the three factors.
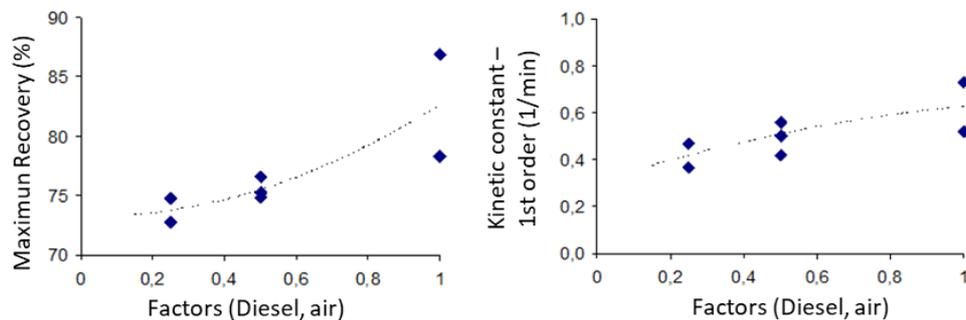

Fig. 12. Correlation between kinetic parameters and factors

Regarding to Klimpel model, for the kinetic constant, the diesel concentration turned out to be the most influential factor, and for the maximum recovery, it was the diesel and aeration interaction. Diesel concentration was the factor which most influenced the flotation kinetics of organic.

3.6. Organic elevation mechanisms

As a result of the previous analysis, it was established that the organic flotation differs from the mineral flotation case; this is because a higher concentration of organic in the flotation tests generated a greater variation of the kinetic constant, an aspect which does not occur in the case of flotation of mineral elements.

This difference can be explained because of: Since the transportation of the organic element to the surface, two mechanisms or kinetics are acting. The first corresponds to the elevation of the organic as a product of its adhesion with the rising bubble. The second one is due to the characteristic of the organic in which its density is lower compared to the water; therefore, that allows it to rise naturally and, in short, the flotation of the organic responds differently during this implementation process.

The phenomenon was notorious in the section and/or initial curve, between intervals of 0 to 5 minutes, which could also be observed graphically, as shown in Fig. 13. The experimental curve represents a linear trend, which differs from mineral flotation models, where an exponential-type plot is obtained.

This section is like zero order mathematic reaction, in which $dc/dt = -k$.

By last, complementary to the above, in Fig. 14, a diagram of the flotation cell is shown, indicating the collection process of organic particles and the ascent to the foam zone. During the collection stage,

the occurrence of a series of phenomena or alternatives in which the organic rises to the foam zone is inferred.

As main cases, three of these, which appear in the diagram, are summarized. Regarding this, some assumptions can be made, such as indicate that, initially, the rise of organic occurs due to these three causes and, in a subsequent period, where the curve becomes more exponential from 4 to 5 minutes, occurs an elevation of organic, as indicated in cases (1) y (2) in which bubbles with organic adhering to particles ascend. It is, specifically, at this point that the flotation process with its characteristics begins to occur.

Thus, the flotation process applied to the organic requires a detailed control regarding these phenomena or characteristics of this element.
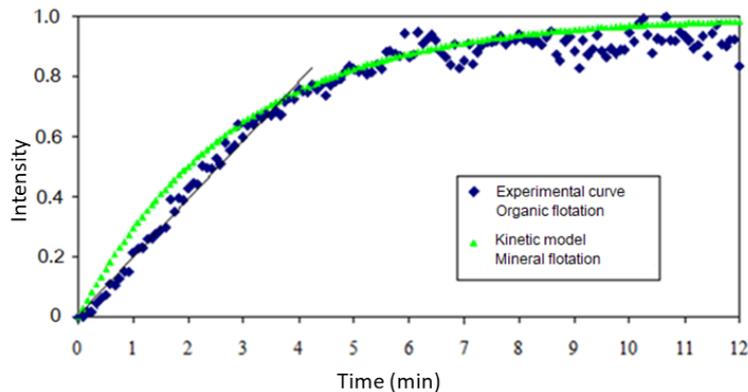

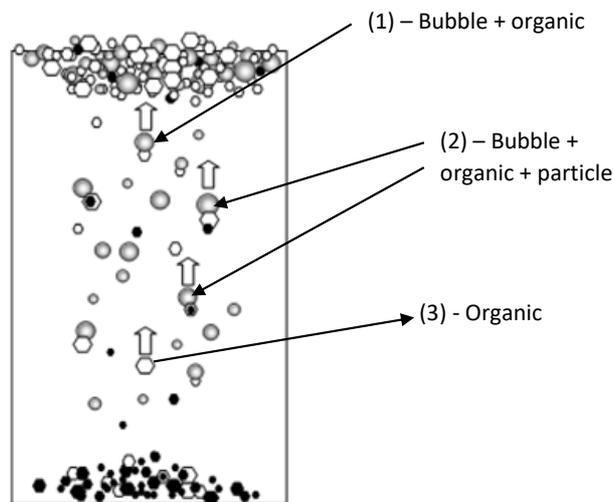Fig. 13. Exponential curve and experimental data

Fig. 14. Elevation of organic cases

4. Conclusions

Experimental evaluations of the flotation process as an alternative technology for removing organic contaminants from soil. The following conclusions can be drawn from the results obtained:

Development of a Novel Methodology: A methodology was developed to distinguish kinetic flotation in laboratory cells by analyzing pulp intensity variations resulting from hydrocarbon removal. This innovative technique has proven to be a practical alternative for studying flotation kinetics in systems where colour variation is a key factor.

Kinetics Modeling: Comparisons of various kinetics models, including first order, Kelsall, and Klimpel, revealed that the first order model provided the best fit to the experimental data. However, it is important to note that a linear behaviour was observed in the kinetics, suggesting characteristics of zero-order kinetics. This phenomenon suggests that the observed response results from the combined removal of droplets carried by bubbles and naturally rising droplets due to differences in densities.

Applicability of Kinetics Models: Our findings also indicate that kinetics models are more suitable for mineral ore flotation than organic flotation. This observation is supported by the linear trend observed in the initial section of the curves, up to 5 minutes, which contrasts with the exponential trend typically seen in mineral ore flotation.

Kinetic Constants and Recovery: The kinetic constants and maximum recovery values of all eight tests conducted achieved extraction rates of over 75% of organic contaminants from the soil within 12 minutes.

In summary, the study underscores the potential of the flotation process for effectively removing organic contaminants from soil. We have developed a valuable methodology for studying flotation kinetics, with practical implications for systems exhibiting colour variation. Additionally, our observations regarding the kinetics models suggest the need for tailored approaches in organic flotation compared to metal flotation. The promising results from our tests demonstrate the rapid and efficient removal of organic contaminants, highlighting the applicability of this method for soil remediation purposes.

Acknowledgments

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