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Theoretical validation model for the treatment of petroleum wastewater using an electron beam

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ABSTRACT

The process of purification of petroleum wastewater by electron beam irradiation using a theoretical model that describes the reactive species generated during irradiation and their reactions with the contaminating substances was analyzed. For the irradiation process, a linear electron accelerator with absorbed doses of 18 and 23 kGy was used on 10 L samples of oil wastewater. The parameters analyzed were: COD, BOD, pH, dissolved oxygen, electrical conductivity, barium ion, sulfates, and calcium ion. The main results show that the purification through the use of electron beam allows the elimination of contaminants such as: COD, BOD, electrical conductivity, barium ion, and calcium ion in a percentages ranging between 16 and 80%. The higher the absorbed dose, the greater the purification percentages of these contaminants. Sulfates, as well as suspended solids in the sample, have contradictory behaviors. After irradiation at the higher absorbed dose (23 kGy), the initially measured values increase. This could be due to a large evaporation of water from the residual water sample.

Keywords: electron beam, wastewater, chemical oxygen demand, barium ion, calcium ion.

INTRODUCTION

Accelerated oxidation in wastewater is the process by which the rapid oxidation of polluting substances occurs; this type of process is also known as advanced oxidation processes (AOPs). These processes are based on the in-situ generation of chemical species with a high oxidation power, such as hydroxyl radicals (OH)-, a non-selective chemical oxidant with a high oxidation potential capable of reacting rapidly with many organic compounds (constant kinetics of the order of 10^6–10^9 $M^{-1}s^{-1}$) (Primo, 2008). The oxidizing species is generated by the direct action of accelerated electrons upon impact with contaminated water, thus forming excited forms and radical species. The free radicals formed oxidize the organic matter (Von, 2007).

Forero, Ortiz and Rios (2005) explain that the main objectives of the application of advanced oxidation processes are: (a) mineralization of contaminants, especially recalcitrant organic ones, until their complete transformation into carbon dioxide, water and inorganic anions, avoiding the formation of byproducts or waste (destructive processes), (b) degradation of organic contaminants into more biodegradable and less toxic compounds, (c) color and odor removal, (d) disinfection.

In this way, when a material is subjected to a source of ionizing radiation, an energy transfer occurs from the source to the exposed system, which will undergo modifications mainly due to the phenomena of excitation and ionization (Cueva, 2001). Charged particles such as electrons are slowed down by the material exposed to radiation and lose their energy through interactions with the irradiated sample (Cherry et al., 2012).

The fraction of energy that is transmitted to the substance from the ionizing particles, that considers linear transfer coefficient (μ_{tr}) and mass per unit area (Δx). The mass energy transfer coefficient considers:

$$\frac{\mu_{tr}}{\rho} = \frac{1}{\rho E}\frac{dE}{dx} \quad (1)$$

where: dx – distance traversed in the middle;
ρ – density of the medium.

It is necessary to know the amount of energy deposited by ionizing radiation in a known volume of the material, absorbed dose. Radiation energy deposited on water is given by the following relationship:

$$Absorbed\ dose\ (D) = \frac{dE}{dm}$$

where: dE – the average energy imparted by the electron beam radiation; dm – amount of water mass; 1 Gray (Gy) is the absorbed dose (1 Gy= 1 J/kg).

The variations in the concentration of organic contaminants in the oil wastewater make subsequent analyses difficult; for this reason, the fixed operating conditions of the accelerator were maintained as shown in Table 1 (Ramirez et al., 1997; Posso, 2001). Table 1 shows the general data of the linear accelerator.

MATERIALS AND METHODS

The linear accelerator delivers pulses of 1 to 5 μs in duration for a repetition of 1 to 500 pulses/s (Posso, 2001), and is part of the Nuclear Sciences Department of the National Polytechnic School (EPN). The final voltage or energy of the accelerated electron appears as a direct current potential in the accelerator microwave.

Table 1. General data of the linear accelerator

Variables	Range
Direction of the Irradiation window	Vertical
Electron pulse duration at 0.5 amplitude	5 μs
Pulse width at 200 Hz	0,790 A
Electron beam frequency	100–200 Hz
Electron beam current at 6 MeV	0,9 A
Potency	0.2–5.6 kW
Sweep length	33–40 cm
Sweep magnet current	34–36 μA
Beam sweep width at 42 μA	40 cm
Electron beam deflection magneto current	30 A
Optimum beam penetration thickness in water	2.5 cm
Maximum water penetration thickness	3.2 cm
Energy as a function of the magnet	7.1 Mev

In the linear accelerator, electrons are emitted by heating the cathode filament (GUN) by AC current and collimated by traveling within a vacuum structure under the action of the vector component of the microwave field. A high vacuum of 10^{-5} to 10^{-8} mmHg is necessary to guarantee that there are no deviations of the beam when colliding with air molecules. To achieve a high vacuum, two or three ion pumps and a mechanical pump are required (Cordero, 2001).

The microwave is generated in the magnetron, which must also reach a high vacuum, which is done by a small ion pump placed inside the accelerator structure. On the outside and completely surrounding the magnetron is a circular solenoid powered by direct current that produces an axial magnetic field.

The electron accelerator has two acceleration stages: (1) electrostatic, using a pulsating potential difference that comes from the modulator; (2) to control the final energy of the electrons by varying the voltage between the cathode and the grid (GUN). Generally, this voltage is of the order of 25 to 30 kV. At this stage, electrons can reach a speed of 2/3 the speed of light. Under these conditions, the electrons are captured by the microwave, which must have a speed lower than that of light. This is achieved by means of rings specially arranged in the waveguide. Simultaneously with the GUN modulator, high pulses are injected into the magnetron cathode. voltage (45 kV) and a short duration of 5 μs to generate a high-frequency 1885 MHz microwave that travels through a rectangular waveguide containing compressed air at 5 atm, with a small phase difference between the pulses of the modulator of the GUN and microwave pulses (Armas, 2006).

This study was conceived as an exploratory assessment of electron beam irradiation for the treatment of petroleum wastewater. Owing to logistical constraints related to sample collection, transport, and access to the irradiation facility, a single experimental irradiation run was performed for each selected absorbed dose. Accordingly, the results are interpreted as preliminary evidence of treatment performance and mechanistic behavior, rather than as a statistically validated dose-response assessment.

Theoretical model for validation of results

Chemical oxygen demand and biochemical oxygen demand (COD and BOD)

To describe the results, it is necessary to understand the primary effects of the electron beam on water. The following equations show the successive processes after applying electron radiation to the oil wastewater samples (OIEA, 2004):

$$H_2O \rightarrow^{rad} H_2O^* \text{ (excited)} \quad (2)$$

$$H_2O^* \rightarrow H^{\cdot} + HO^{\cdot} \quad (3)$$

$$H_2O^* \rightarrow H_2O^+ + e^- \quad (4)$$

where: $H^{\cdot}$ y $HO^{\cdot}$ (free radicals),
H_2O^+ (ionization).

Domènech, et al. (2001) pointed that the aqueous electron e^-_{aq} is formed as follows:

$$e^- + n\,H_2O \rightarrow e^-_{aq} \quad (5)$$

Where the aqueous electron interacts with itself and with the hydronium ions present in the water, forming hydrogen and hydrogen free radicals:

$$e^-_{aq} + e^-_{aq} \rightarrow^{H_2O} H_2 + 2OH^- aq \quad (6)$$

$$e^-_{aq} + H_3O^+ \rightarrow H_2O + H^{\cdot} \quad (7)$$

Hydrogen free radicals, H·, form hydrogen molecules through the process of free radical recombination. Another primary effect of the electron beam on water molecules is:

$$H_2O \rightarrow^{rad} H_2O^{\cdot +} + e^- \text{ (Ionization)} \quad (8)$$

$$H_2O \rightarrow^{rad} H_2O^{\cdot} \quad (9)$$

The $H_2O^{\cdot +}$ radical ion or the H_2O^+ ion reacts with the water molecule to form $HO^{\cdot}$ radicals:

$$H_2O^{\cdot +} + H_2O \rightarrow H_3O^+ + OH^{\cdot} \quad (10)$$

$$H_2O^+ + H_2O \rightarrow H_3O^+ + HO^{\cdot} \quad (11)$$

The $HO^{\cdot}$ radicals that can recombine with each other to form hydrogen peroxide H_2O_2, while the radicals $H^{\cdot}$ and $HO^{\cdot}$ can combine to form water.The main elements that are finally produced by ionizing radiation are: $H^{\cdot}$, $HO^{\cdot}$, and e^-_{aq}, These products can produce oxidation and reduction reactions, depending on the pH of the water, mainly organic and inorganic components.

The primary species $H^{\cdot}$, $HO^{\cdot}$, and e^-_{aq}, are highly reactive and can alter organic molecules, forming R· radicals and hydrogen gas molecules, CO_2, fragments of alcohol molecules, esters, and hydrocarbons of up to 2 carbons (UNDP, 1993). The breaking of bonds of organic matter, as well as its subsequent precipitation or transformation into CO_2, allows the reduction of organic matter (Duarte et al., 2004).

pH

In order to explain the evolution of the pH in the sample, the equation for the radiolysis of water must be remembered (Cueva, 2001; Criquet and Leitner, 2015; Wojnarovits and Takacs, 2008):

$$H_2O \xrightarrow{rad} 2{,}7OH^{\circ} + 2.6\,e^-aq + 0{,}6\,H + \\ + 2{,}6\,H_3O^+ + 0{,}45\,H_2 + 0{,}7\,H_2O_2 \quad (12)$$

It can be determined that the greatest production of primary species is that of being a free radical which will react with organic compounds, however depending on the characteristics of the sample (acidic or basic) the radicals will increase the pH values.

Dissolved oxygen

The irradiation of electrons in the wastewater sample forms aqueous electrons, the aqueous electrons react with oxygen in the presence of hydrogen by the following equation (Giraldo et al., 2004):

$$e^-aq\,(H) + 2O_2 \rightarrow O_2^-(HO_2) \quad (13)$$

$$O_2^-\,Oxygen + e^-aq \quad (14)$$

$$O_2^- + 2H^+ \rightarrow H_2O_2 \quad (15)$$

A decrease in the concentration of dissolved oxygen in the residual water sample is expected; however, it is possible that when irradiating with the electron beam, the decrease in contaminants causes an increase in the partial pressure of oxygen, which causes the concentration gradient between the partial pressure of atmospheric oxygen and that of the sample to decrease, this can allow atmospheric oxygen to enter the residual water (Muedas, 2021).

Electric conductivity

The electrical conductivity in a wastewater sample is related to the ability of the water to conduct electrical current, and it is clear that the

electrical conductivity depends directly on the concentration of ions present in the sample (Ba^{2+}, Ca^{2+}, Mg^{2+}), that is, the lower the concentration of Ions, the less conductivity.

The irradiation of the electron beam on the wastewater sample allows anions to react with cations, and therefore the concentration of ions decreases; in this way, the electrical conductivity should be lower after irradiation.

Barium ion

The theoretical analysis of the ionizing radiation on the barium ion allows determining approximately what can be obtained as a result of the application of electrons to the wastewater. The Barium ion is a cation that is found in different concentrations in the wastewater sample. Its +2 oxidation state maintains it until an anion can react with it to form a compound. Ionizing radiation on the barium ion can fundamentally produce the following (Roth et al., 2008):

$$Ba^{+2} \xrightarrow{rad} Ba^{+2} \quad (16)$$

$$Ba^{+2} \xrightarrow{rad} (Ba^{+2})^{*} \quad (17)$$

$$(Ba^{+2})^{*} + ((XY)^{=})^{*} \rightarrow Ba(XY) + hf_1 \quad (18)$$

where: $(Ba^{+2})^{*}$ excited barium cation;
$((XY^{=})^{*}$ excited anion, present in the wastewater sample;
hf_1 – photon that results from the combination of ions, where h is Planck's constant, f_1 is the frequency of the emitted photon.

Equation 17 shows the barium ion, which is measured in the sample and is its toxic agent. The radiation produces an excitation on the ion; it cannot ionize it because it is in its maximum oxidation state, therefore the radiation only transfers enough energy to excite it, there will be no dissociation of the ion or chemical changes in its structure.

The excited ion (18) remains unstable until a recombination of ions (anion-cation) occurs. The molecule formed will depend on the anions (Equation 18) present in the sample. In the final reaction, the excess energy is released as a photon.

According to the previous considerations, a significant decrease in the barium ion when the electron beam is applied to the sample is estimated.

Sulfates

The concentration of sulfate ions determines the reactions that can occur when treating wastewater. The sulfate ion with oxidation state -2 will remain as an ion until its initial thermodynamic conditions are altered (Roth et al., 2008). These conditions will change when the irradiation is applied to the sample, forming some salt that can then precipitate. The reactions between the Sulfate anion and a cation are the following:

$$SO_4^{-2} \xrightarrow{rad} SO_4^{-2} \quad (19)$$

$$SO_4^{-2} \xrightarrow{rad} (SO_4^{-2})^{*} \quad (20)$$

$$(SO_4^{-2})^{*} + (R^{+2})^{*} \rightarrow SO_4R + hf_2 \quad (21)$$

where: SO_4^{-2} excited sulfate anion; $(R^{+2})^{*}$ excited cation, present in the wastewater sample.

The sulfate ion concentration will decrease proportionally when reacting with the cations that exist in the sample; however, if a large evaporation of water occurs due to irradiation, it is likely that the sulfate measurement will be even higher than at the beginning.

Calcium ion

The calcium ion is also a cation, with an oxidation state +2; its oxidation state, as for the barium ion, will be affected by the electron beam, exciting it to then recombine with the anions present in the wastewater. The reactions are similar to those obtained for the barium ion.

$$Ca^{+2} \xrightarrow{rad} Ca^{+2} \quad (22)$$

$$Ca^{+2} \xrightarrow{rad} (Ca^{+2})^{*} \quad (23)$$

$$(Ca^{+2})^{*} + ((QZ)^{=})^{*} \rightarrow Ca(QZ) + hf_3 \quad (24)$$

where: $(Ca^{+2})^{*}$ excited barium cation;
$((QZ)^{=})^{*}$ excited anion, present in the wastewater sample; hf_3 –photon that results from the combination of ions, where h is Planck's constant, f_3 – the frequency of the emitted photon.

Equation 22 shows the calcium ion, the radiation produces an excitation on the ion, it cannot ionize it due to its maximum oxidation state, therefore, the radiation only transfers enough energy to excite it, there will be no dissociation of the ion or chemical changes in its structure.

The excited ion (23) remains unstable until a recombination of ions occurs. The molecule formed will depend on the anions (24) present in the sample. In the final reaction, the excess energy is released in the form of a photon. Under these considerations, a decrease in the concentration of the calcium ion in the sample is expected, as for the barium ion.

RESULTS AND DISCUSSION

Waste oil water samples treated by electron irradiation

The parameters of the electron accelerator remain constant as previously established, while the absorbed dose delivered to the samples was varied. Absorbed doses of 23 kGy and 18 kGy are used for this work. The first sample was irradiated at an absorbed dose of 23 kGy, the results are in Table 2.

The best results for COD and BOD_5 removal are obtained with an irradiation dose of 23 kGy, and are approximately 50%, the removal of Calcium Ion and Barium Ion also present higher removal percentages at this absorbed dose, with 25% and 80% respectively. On the other hand, some parameters increase significantly, such as DO increases in 900% and Sulfate ion increases in 100%.

Figure 1 shows the removal percentages obtained for COD, BOD_5, electrical conductivity, Ba^{2+} and Ca^{2+} after electron beam irradiation at absorbed doses of 18 and 23 kGy. Although the results suggest that the higher absorbed dose improved the removal of several parameters, the initial wastewater composition differed between samples. Therefore, these results should be interpreted as preliminary evidence, and a broader replicated dose-response study is required to confirm this trend. Suspended solids and sulfates showed a different behavior and are therefore discussed separately.

Table 2. Data obtained from the oil wastewater sample

Parameters	Irradiation at 23 kGy	
	Water sample (no treatment)	Water sample (irradiated)
COD (mg O_2/l)	2252	1081
BOD_5 (mg O_2/l)	1000	480
pH	6.41	6.56
Dissolved oxygen (mg O_2/l)	0,5	5,0
Suspended solids (mg/l)	138	171
Conductivity (µS/cm)	3300	1350
Barium (mg Ba^{2+}/l)	40	8
Sulfates (mg SO_4^{2-}/l)	400	800
Calcium (mg Ca^{2+}/l)	300	224

Note: For the second wastewater sample, the irradiation dose was 18 kGy, the results are shown in Table 3.

Table 3. Data obtained from an oil wastewater sample

Parameters	Irradiation at 18 kGy	
	Water sample (no treatment)	Water sample (irradiated)
COD (mg O_2/l)	750	440
BOD_5 (mg O_2/l)	433	280
pH	6.85	6.96
Dissolved oxygen (mg O_2/l)	2	4.5
Suspended solids (mg/l)	158	112
Conductivity (µS/cm)	1220	750
Barium (mg Ba^{2+}/l)	50	15
Sulfates (mg SO_4^{2-}/l)	645	520
Calcium (mg Ca^{2+}/l)	225	189

Note: Table 4 summarizes the obtained results by irradiating the samples using the electron beam.

Table 4. Results of electron beam irradiation on the residual oil water sample

Irradiation	Water sample	COD (mg/L)	BOD_5 (mg/L)	pH	DO (mg/L)	Suspended solids (mg/L)	Conductivity (µs/cm)	Ba^{+2} (mg/L)	SO_4^{-2} (mg/L)	Ca^{+2} (mg/L)
23 kGy	No treatment	2252	1000	6.41	0.5	138	3300	40	400	300
	Irradiated	1081	480	6.56	5.0	171	1350	8	800	224
18 kGy	No treatment	750	433	6.85	2	158	1220	50	645	225
	Irradiated	440	280	6.96	4.5	112	750	15	520	189

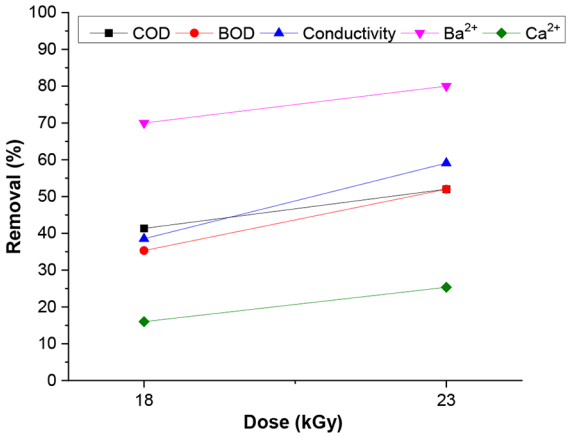

Figure 1. Percentages of contaminant removal from two absorbed doses of electron beam irradiation (18 and 23 kGy)

Chemical oxygen demand and biochemical oxygen demand (COD and BOD_5)

The decrease in organic matter, expressed as COD and BOD_5, was higher at the absorbed dose of 23 kGy under the tested conditions, with removal percentages close to 52%

The highly reactive primary species $H^{\cdot}$, $HO^{\cdot}$ and are produced in greater quantities when the absorbed doses are higher, which generates the breaking of bonds of organic matter that then precipitates or is transformed into CO_2.

For an irradiation of 18 *kGy,* the percentage in which organic matter decreases is 41.33% for COD and 35.3% in the case of BOD_5; the elimination of percentage organic matter is lower due to the lower generation of primary reactive species (Duarte et al., 2000).

Although the generation of reactive species is related to the absorbed dose delivered to the sample, treatment efficiency also depends on the concentration and composition of the organic compounds present in the wastewater. Therefore, even when the absorbed dose is increased, the availability of reactive species and their effective interaction with the contaminants may be limited by the characteristics of the sample matrix (Gryczka et al., 2021).

pH

The evolution of the pH in the irradiated samples is as expected according to the radiolysis equation:

$$H_2O \xrightarrow{rad} 2.7OH^{\circ} + 2.6\, e^{-}aq + 0.6\, H + \\ + 2.6\, H_3O^{+} + 0.45\, H_2 + 0.7\, H_2O_2 \qquad (25)$$

In which the primary species OH° are highly reactive and produce a small percentage increase in pH. Thus, in the sample irradiated with 23 kGy, the pH variation is 2.34% (from 6.41 to 6.56) while in the sample irradiated with 18 kGy it is 1.6% (from 6.85 to 6.96), the samples remain acidic, although close to pH = 7 and with insignificant variations (Cueva, 2001).

Dissolved oxygen

The previously described equations implied a decrease in dissolved oxygen in the irradiated sample; however, the results obtained allow us to determine an increase in oxygen in the oil wastewater. The first sample irradiated at 23 kGy increases by 900% (from 0.5 to 5 mg/L), while for the second test at 18 kGy it increases by 125% (from 2 to 4.5 mg/L). The proposed model determined a decrease in dissolved oxygen, but in practice, this does not happen. Thus, after irradiation with the electron beam, a decrease in contaminants may cause an increase in the partial pressure of oxygen, which decreases the concentration gradient between the partial pressure of atmospheric oxygen and that of the sample. Then, the consequence is the entry of atmospheric oxygen into the oil wastewater (Sun, Zhang, and Quan, 2008).

Electric conductivity

The irradiation of the electron beam allows anions to react with cations and therefore the concentration of ions in the sample is lower, in this way the electrical conductivity decreases by 59.1% (from 3300 to 1350 μs/cm) when it is irradiated at 23 kGy, while if the sample receives irradiation of 18 kGy it decreases by 38.52% (from 1220 to 750 μs/cm).

The decrease in electrical conductivity after irradiation is mainly associated with changes in the ionic composition of the wastewater. Under the conditions evaluated, electron beam irradiation likely promoted reactions, recombination, or precipitation of dissolved ions, reducing the concentration of charge carriers in the sample (Chaudhary et al., 2015).

Barium ion

For acidic wastewater such as the samples analyzed, the removal capacity of the Barium ion increases, considering that the sulfate group is a chelating group of barium, the high content of the sulfate group benefits the absorption of the barium ion (Kang et al., 2017). The results show a decrease of 80% for the most energetic irradiation, going from 40 to 8 mg/L, while for the irradiation with the lower absorbed dose, the percentage decrease is 70%, going from 50 to 15 mg/L.

Sulfates – suspended solids

Like the hydroxyl radical, sulfate radicals are highly reactive species with a short lifespan; sulfate radicals tend to remove electrons from organic compounds. Molecules that are subsequently transformed into organic cations (Deng, Zhao, 2015).

According to the theoretical model proposed, a decrease in sulfate ion with oxidation state -2 was expected when reacting with the cations that exist in the sample; a similar decrease was expected with respect to the suspended solids. However, the results obtained for two of the two samples seem to be contradictory. For the first irradiation at 23 kGy the sulfates increase by 100% (from 400 to 800 mg/L), and at the same time, the suspended solids increase in the sample by 23.91% (from 138 to 171 mg/L). On the contrary, for the second sample at 18 kGy the sulfates also decrease the suspended solids; sulfates decrease by 19.37% (from 645 to 520 mg/L), and suspended solids decrease by 29.11% (from 158 to 112 mg/L).

Generally, the irradiation process of the electron beam is absorbed by the water of the sample and not by the solutes present (Chmielewski, 2005). This implies that the greater the absorbed dose with the smaller amount of solutes, the greater the evaporation of water and volatile compounds, which affects the subsequent measurement of suspended solids and sulfates, and that would explain the contradictory behavior at 23 kGy.

Calcium ion

According to the equations in the theoretical model, in the calcium ion, its oxidation state will be affected by the electron beam, exciting it and then recombining with the anions present in the residual water (Frank, 1995).

After irradiation at 23 kGy, the calcium ion decreases by 25.33% (from 300 to 224 mg/L), the results for the sample irradiated at 18 kGy are similar; there is a decrease of 16% (from 225 to 189 mg/L).

CONCLUSIONS

The present study evaluated the use of electron beam irradiation for the treatment of petroleum wastewater and proposed a theoretical framework to describe the reactive species generated during water radiolysis and their interactions with selected organic and inorganic contaminants.

Under the experimental conditions evaluated, electron beam irradiation promoted a clear reduction in several wastewater quality parameters, including COD, BOD_5, electrical conductivity, Ba^{2+} and Ca^{2+}. The highest removal efficiency was observed for Ba^{2+}, while COD and BOD_5 showed moderate but relevant decreases, indicating the potential of this technology for reducing both organic load and selected ionic contaminants in petroleum wastewater.

The proposed theoretical model provides a qualitative explanation for the observed decrease in COD, BOD_5, electrical conductivity, $Ba^{2+,}$ and Ca^{2+}, mainly through the formation of reactive radical species during water radiolysis and their subsequent oxidation, reduction or recombination reactions. However, the model should be

considered a preliminary mechanistic framework rather than a fully predictive quantitative model.

The behavior of dissolved oxygen, sulfates and suspended solids indicates that additional physicochemical processes may occur during irradiation. In particular, oxygen transfer from the surrounding atmosphere, partial water evaporation, oxidation of reduced sulfur species, destabilization of emulsified phases, and changes in the distribution between dissolved and suspended matter may influence the final measured concentrations.

Although electron beam irradiation showed promising results for the treatment of petroleum wastewater, further studies are required to validate the process under replicated experimental conditions, evaluate a wider absorbed-dose range, quantify possible water losses, identify intermediate degradation products, and improve the model for complex parameters such as sulfates and suspended solids.

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