Optimization of persulfate/iron(II)/UV-A irradiation process for the treatment of landfill leachate from Fez City (Morocco)

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Abstract

The objective of this study is to investigate the efficiency of the advanced oxidation process using persulfate ions $S_2O_8^{2-}$ (PS), Fe^{2+} , and UV-A irradiation for the treatment of stabilized leachate from the urban sanitary landfill of Fez city (Morocco). In fact, in the presence of an activating agent, $S_2O_8^{2-}$ generates sulfate radicals ($SO_4^{\cdot-}$) which are powerful oxidizers for organic matter. The effect of reaction time, PS and Fe^{2+} dosage on the raw landfill leachate treatment was evaluated to define the optimum operational conditions according to chemical oxygen demand (COD) and color removal. Hence, the optimization of these process parameters led to a maximal removal of COD (70%) and color (94%) using $S_2O_8^{2-}$ dose = 14,000 mg L⁻¹, Fe^{2+} dose = 1600 mg L⁻¹, and pH = 3 under 60 min of UV-A irradiation. The application of UV-A had a positive effect on the treatment efficiency, PS/ Fe^{2+} /UV-A has indeed enhanced COD removal by (+ 10%) compared with PS/ Fe^{2+} only. The results of this study showed that the oxidative system (PS/ Fe^{2+} /UV-A) can achieve a high level of COD and color removal from landfill leachate. Consequently, the high efficiency, the easy use, and the fast mechanism could make this system as a promising and alternative process for landfill leachate and wastewater treatment.

Keywords Chemical oxidation · Activated-persulfate · UV-A · Leachate treatment · Organic pollutants

1 Introduction

Landfill leachate is defined as the effluent generated by rainwater percolation through landfilled solid waste. Thus, leachate is bad-smelling, blackish, and loaded with various and complex pollutants. This justifies the considerable attention paid to solve this issue through several studies developing different treatment processes applied to real or synthetic effluents [1–5].

In the last few years there has been a growing interest in advanced oxidation processes (AOPs) as efficient methods for pollutants removal from aqueous phases [6]. For instance, Fenton, photo-Fenton [7], Fenton-Like [8], ozonation [9] and UV irradiations [10] processes, mostly applied, are based on the generation of hydroxyl radicals

($\cdot OH$) which has a high standard oxidation potential ($E^\circ \approx 2.8$ V at 25 °C) [6].

In addition, persulfate ($S_2O_8^{2-}$) is one of the strong oxidants widely used in the degradation of various organic contaminants from the water and wastewater. In fact, despite its disadvantages, such as the narrow working pH range, the high cost, the production of intermediate pollutants, and the iron sludge production in the case of Fe^{2+} as a catalyst, previous studies have proved that PS was successfully applied in different oxidative systems (Ozone/PS, UV/PS, Microwave/PS, Iron/PS, Aluminum/PS) for COD removal from landfill leachate [11–15]. Actually, this effectiveness is due to its high chemical reactivity, stability, and wide pH range in liquid phases. Moreover, it is known by a high standard oxidation potential ($E^\circ = 2.01$ V) [16, 17] and

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ability to initiate sulfate radical ($\text{SO}_4^{\cdot-}$) which has a high oxidizer effect for organic compounds degradation because of its important oxidation potential ($E^\circ = 2.7 \text{ V}$) [18]. These sulfate radicals are generated throughout persulfate activation which initiated by various processes such as heat energy, exposition to UV irradiation or addition of transition metals [19–21], according to the following Eqs. (1–3):

In Morocco, landfill leachate issues are gaining increasing importance, where dozens of research works have been conducted on the characterization and the treatment of this effluent sampled from different municipal landfills of Moroccan cities. However, the majority of these studies applied mainly coagulation-flocculation and biological as processes treatment [22–24]. To our knowledge, the application of PS/ Fe^{2+} /UV for the treatment of landfill leachates has not been reported elsewhere. Hence, the current work is considered as the first study that applies simultaneously PS, Fe^{2+} and UV irradiations in landfill leachate treatment.

Therefore, this study aims (a) to compare the application $\text{S}_2\text{O}_8^{2-}/\text{Fe}^{2+}$ and $\text{S}_2\text{O}_8^{2-}/\text{Fe}^{2+}/\text{UV-A}$ systems for the treatment of Fez city landfill leachate with respect to COD and color removal and (b) to evaluate the effects of PS, iron dosages and reaction time on COD and color elimination.

2 Experiments

2.1 Chemicals and reagents

Potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$), ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), sulfuric acid (H_2SO_4) and sodium hydroxide (NaOH) were purchased from Sigma-Aldrich chemicals (St. Louis, Missouri, USA).

2.2 Leachate sampling and characterization

The leachate used in this study was obtained from municipal solid waste landfill (MSWL) located at 11 km from Fez city, Morocco. This landfill receives 1000 tons of solid waste per year and generates about 350–450 m^3 of leachate per day. The raw leachate was sampled in opaque plastic bottles, transported immediately to the laboratory, and stored in the dark at 4 °C in order to prevent any biological or chemical reactions. Thereafter, the physical–chemical

parameters of the sample were measured: temperature, pH, electrical conductivity, turbidity, TSS (total suspended solid), COD, BOD_5 (biological oxygen demand), nitrates, nitrites, sulfates, in accordance with the standard methods previously used in a published study [7]. The main characteristic parameters of landfill leachate are shown in Table 1. It is noticed that the studied leachate is highly loaded with different pollutants, expressed by high values of TSS, turbidity, COD, BOD_5 , and color number. Moreover, the pH value (> 7.5) and BOD_5/COD ratio (< 0.1) reveal that this sample has a low biodegradability and could be designated as stabilized leachate. Consequently, the refractory substances of this leachate could be effectively removed using physical–chemical processes [25, 26].

2.3 Procedure of leachate treatment

Potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$) was used as a source of $\text{S}_2\text{O}_8^{2-}$, while Fe^{2+} ions were provided by ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), and pH leachate was adjusted using 2 M H_2SO_4 or 2 M NaOH .

The leachate treatment tests were conducted at ambient temperature in batch mode, under magnetic stirring of $\text{K}_2\text{S}_2\text{O}_8$ and FeSO_4 amounts added to a volume of raw leachate. Firstly, the preliminary experiments were carried out in a cylindrical pyrex photoreactor in order to fix the initial pH of leachate and to determine the range of $\text{S}_2\text{O}_8^{2-}$ and Fe^{2+} dosages. Thereafter, the contact time effect

Table 1 Physico-chemical characteristics of landfill leachate from Fez city

Parameters	Unit	Value
Electrical conductivity	mS cm^{-1}	34
T	°C	24
pH	–	8
TSS	mg L^{-1}	14,000
Turbidity	NTU	218
COD	$\text{mg O}_2 \text{ L}^{-1}$	5198
BOD_5	$\text{mg O}_2 \text{ L}^{-1}$	500
BOD_5/COD	–	0.09
Color number	–	6.52
SO_4^{2-}	mg L^{-1}	140
NO_3^-	mg L^{-1}	8.13
NO_2^-	mg L^{-1}	7.15
Cl^-	mg L^{-1}	6845
SO_4^{2-}	mg L^{-1}	140
Cr	mg Cr L^{-1}	1.74
Cu	mg Cu L^{-1}	0.05
Fe	mg Fe L^{-1}	1.83
Mn	mg Mn L^{-1}	0.043
Zn	mg Zn L^{-1}	0.2

was investigated under fixed values of pH, $S_2O_8^{2-}$ and Fe^{2+} concentration. A volume of 5 mL of the sample was collected at regular time intervals of the treatment and centrifuged for 10 min at 5000 rpm for the UV–visible spectrophotometry analysis and COD measurements. Afterwards, the effect of $S_2O_8^{2-}$ dosage was studied at optimal pH and contact time. Finally, Fe^{2+} dosage was selected under the optimal conditions of pH, contact time and $S_2O_8^{2-}$ concentration. The optimal parameters values were obtained based on the best performances in terms of COD removal and UV–Visible spectra variation, which were measured for each sample at different points of the parameter range.

Nevertheless, to study the effect of UV-A irradiations, the same procedure was followed under the light of three UV-A lamps of (20 W, 365 nm) in the same photoreactor (Fig. 1) [5].

COD removal was calculated using the following formula:

$$\text{COD removal(\%)} = \frac{\text{COD}_i - \text{COD}_f}{\text{COD}_i} \times 100 \quad (4)$$

where COD_i and COD_f are COD of raw and treated leachate, respectively.

Color number (CN) was calculated using a formula related to the measurement of absorbance values at three wavelengths 436 nm, 525 nm, and 620 nm (5) [27].

$$\text{CN} = \frac{\text{SAC}_{436}^2 + \text{SAC}_{525}^2 + \text{SAC}_{620}^2}{\text{SAC}_{436} + \text{SAC}_{525} + \text{SAC}_{620}} \quad (5)$$

$$\text{SAC}_i = \frac{\text{Abs}_i}{x} \quad (6)$$

With SAC is the spectral absorption coefficient at each wavelength, and x is the thickness of the used cell.

Fig. 1 Schematic of photoreactor with suspended UV-A lamps

3 Results and discussion

In order to evaluate the efficiency of PS/ Fe^{2+} and PS/ Fe^{2+} /UV-A treatments, the reaction time effect was initially investigated. Afterwards, COD removal and UV–Vis spectrum were carried out as a function of PS and Fe^{2+} dosage under optimal values of pH and reaction time. It should be noted that preliminary tests were performed with the aim of choosing the PS and Fe^{2+} dose range and fixing pH at 3.

3.1 Contact time effect

The reaction time was examined from 5 to 120 min at fixed pH, PS, and Fe^{2+} dosage (pH = 3; [PS] = 3000 mg L⁻¹; [Fe^{2+}] = 1000 mg L⁻¹). Figure 2 illustrates the COD removal evolution depending on reaction time, under fixed amounts of PS, Fe^{2+} , and pH value. Curve displayed in Fig. 2 indicates that the COD removal increases rapidly with the reaction time, where 46% of COD was reduced in the first 5 min. After that, the efficiency gradually becomes slow and reaches the maximal removal ($\approx 62\%$) after 60 min. These results could be explained by the consumption of $S_2O_8^{2-}$ and Fe^{2+} with reaction time [13, 28] and allow to choose 60 min as the optimal contact time, and that was selected for studying the following parameters effect.

In fact, similar studies revealed that the majority of pollutants are reduced in the first minutes of reaction [13, 29], because of the availability of sulfate radicals produced during the reaction (7) that oxidizes organic

Fig. 2 COD removal depending on contact time: [PS] = 3000 mg L⁻¹; [Fe^{2+}] = 1000 mg L⁻¹; solution pH = 3

contaminants [30, 31]. Furthermore, the organic matter could be degraded by hydroxyl radicals ($\cdot\text{OH}$) which are produced through the reaction of $\text{SO}_4^{\cdot-}$ with H_2O and/or OH^- according to the reactions (8) and (9) [32–35].

Fig. 3 Effect of PS dosage on COD removal (with and without UV-A irradiation): $[\text{Fe}^{2+}] = 200 \text{ mg L}^{-1}$; solution pH=3; contact time=60 min

3.2 PS dosage effect

In order to explore the PS dose influence on process efficacy in terms of COD removal (Fig. 3), PS dose was varied between 0 and 55,000 mg L^{-1} . Thus, increasing PS dosage from 0 to 3000 mg L^{-1} enhanced COD removal from 34 to 51% in dark conditions, however, it increased from 42 to 63% with UV-A light.

In fact, several studies have proved that UV-A irradiation enhances the PS decomposition into sulfate radicals (3), which have powerful effects on the organic compounds oxidation [12, 33, 36], following the proposed mechanism presented in Fig. 4.

However, rising the PS dose to 55,000 mg L^{-1} decreased slightly the efficiency of the process to 58% for irradiated sample, though it reached 37% for non-irradiated one. Similarly, several studies indicated that above an optimal persulfate dosage, the sulfate radicals could react with the excess quantity of PS (reaction 10), or they react with themselves (reaction 11), or with the catalyst Fe^{2+} (reaction 12), which inhibits the oxidation of organic compounds [13, 28, 31, 37]. Hence, 1400 mg L^{-1} was fixed as the optimal concentration of persulfate ions.

Figure 5 presents the evolution of UV-Vis spectra of leachate samples according to PS dosages. The results, as shown in Fig. 5, indicate that varying PS dose from 0 to 14,000 mg L^{-1} significantly reduced the absorbance values in the range of 250–300 nm which is characteristic of the presence of organic matter, especially aromatic

Fig. 4 Proposed mechanism for the organic matter degradation using activated Persulfate by Fe^{2+} and UV-A irradiation

Fig. 5 Effect of PS dosage on UV-Vis spectrum (with UV-A irradiation)

compounds, hydrocarbons and humic acids [38, 39]. The decrease of the absorbance values correlates with the variation of COD removal presented in Fig. 3 [40, 41], indicating the evolution of the organic matter degradation at different PS dosages.

3.3 Fe^{2+} dosage effect

After optimizing PS dosage at $14,000 \text{ mg L}^{-1}$, the effect of Fe^{2+} dosage was investigated in a range of $0\text{--}3000 \text{ mg L}^{-1}$ in terms of COD removal (Fig. 6) and UV-Vis spectrum variation (Fig. 7).

Fig. 6 Effect of Fe^{2+} dosage on COD removal (with and without UV-A irradiation): $[\text{PS}] = 14,000 \text{ mg L}^{-1}$; $\text{pH} = 3$; contact time = 60 min

Fig. 7 Effect of Fe^{2+} dosage on UV-Vis spectrum (with UV-A irradiation)

As shown in Fig. 6, increasing the ferrous ions amount has a positive impact on COD reduction for both treatments (with and without UV-A irradiation). For non-irradiated sample, varying Fe^{2+} dose from 0 to 200 mg L^{-1} improved COD removal from 29 to 47%. The same evolution was noticed for the irradiated sample where the process efficacy enhanced from 41 to 61%. Afterwards, an additional COD removal was obtained (+ 10%) when further Fe^{2+} amount was added.

These results are in agreement with similar findings, which proved that under the UV irradiations, persulfate can be promptly transformed into two sulfate radical anions ($\text{SO}_4^{\cdot-}$) following the reaction (3) [42–45].

Moreover, a considerable decrease of the UV-Visible absorbance in the range $250\text{--}300 \text{ nm}$ with increasing the amount of ferrous ions from 0 to 1600 mg L^{-1} , is observed in Fig. 7 which confirms the significant positive effect of Fe^{2+} addition on the process mechanism for the organic matter degradation [46]. Consequently, the optimal Fe^{2+} dosage was fixed at 1600 mg L^{-1} .

Such positive effect of metal catalysts on organic pollutants oxidation has been previously demonstrated [6, 21, 30], especially that Fe^{2+} is an effective PS activator favoring then the production of sulfate radicals following the reaction 12 and presented by the mechanism in Fig. 4. However, exceeding the optimal dosage could negatively affect the treatment efficiency [47, 48], due to the sulfate radicals consumption by the excess of Fe^{2+} ions through (reaction 11).

Fig. 8 COD and color removal depending on different combinations of ([PS] = 14,000 mg L⁻¹; [Fe²⁺] = 1600 mg L⁻¹)

3.4 Optimized conditions

Figure 8 summarizes the COD and color removal efficiencies obtained by different treatment combinations of Fe²⁺, PS and UV. Hence, the following order PS/Fe²⁺/UV > PS/Fe²⁺ > Fe²⁺/UV > PS/UV > Fe²⁺ > PS > UV is noticed in terms of COD removal, while this order PS/Fe²⁺/UV > PS/Fe²⁺ > PS/UV > PS > Fe²⁺ > Fe²⁺/UV > UV was deduced in terms of color removal.

This result showed that the combined system (i.e., PS/Fe²⁺/UV) was efficient for COD and color removal compared with other studied processes.

On the basis of the previous results, it could be concluded that the optimum conditions for the studied landfill leachate treatment are: pH = 3, reaction time = 60 min, [PS] = 14,000 mg L⁻¹ and [Fe²⁺] = 1600 mg L⁻¹ under UV-A irradiations. Hence, a remarkable treatment efficacy is noticed in the image of sample leachate before and after treatment under the optimum operating conditions (Fig. 9).

4 Conclusion

Based on the results of this research work, it was evident that PS/Fe²⁺/UV-A system was successfully applied for removing the chemical oxygen demand (COD) of stabilized leachate. The results revealed that the efficiency of PS/Fe²⁺/UV-A system with respect to the COD removal was influenced by several operational parameters which included the dosage of PS, Fe²⁺, and reaction time. At optimum conditions of PS/Fe²⁺/UV-A and PS/Fe²⁺ processes (14,000 mg L⁻¹ of S₂O₈²⁻, 1600 mg L⁻¹ of Fe²⁺, and pH = 3

Fig. 9 Pictures of raw and treated leachate by PS/Fe²⁺/UV-A process

for 60 min of reaction time), maximum COD removals were found 70% and 59%, respectively. It was also demonstrated that the optimization of each reagent dosage is highly required to avoid the inhibition of sulfate radicals production.

Indeed, the promising findings presented in this paper will provide further studies in order to maximize the treatment performance by testing other activators of persulfate or by coupling this process with other treatments. Especially that landfill leachate is known by its refractory and complex compounds.

Finally, it can be concluded that PS/Fe²⁺/UV system could be considered as a new competitive and efficient process for leachate treatment in Morocco in comparison with other processes treatment previously studied.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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