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Optimization of H_2O_2 and ozone-based advanced oxidation processes as pretreatment of sanitary landfill leachate

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ABSTRACT

The direct discharge of leachate into the environment may have catastrophic effects on both water and soil. In this study, lab tests were performed to compare the efficiency of several single and hybrid advanced oxidation processes (AOPs) in treating landfill leachate in terms of chemical oxygen demand removal percentage (COD RP) and leachate pollution index removal percentage (LPI RP). Conditions were optimized for leachate treatment by ozonation, H_2O_2, and Fenton process as single AOPs and then combined as hybrid techniques (Fenton/O_3 and H_2O_2/O_3) in their optimized conditions. All optimization conditions were performed in terms of COD RP. The results indicated that, for single methods: leachate treated by ozonation process has been optimized at pH 8.4 and 120 min contact time (CT); H_2O_2 at pH 11 and 4 g L^{-1} dose and Fenton process at pH 3.5, $[H_2O_2]$:$[Fe^{+2}]$ ratio = 4:1.4 g L^{-1} and 50 min CT. All used methods are strongly influenced by the pH value of the treated medium. Regarding hybrid methods: The Fenton/O_3 method was observed to have the highest removal efficiency for COD and LPI in acidic medium among all H_2O_2-based AOPs.

Keywords: Advanced oxidation processes; Landfill leachate; Hydrogen peroxide; Fenton process; Ozonation; Hybrid technique

1. Introduction

Leachate is defined as liquid produced mainly by passing percolated precipitant through the open or cap of the closed landfill site. To be more specific, it is a smelly, dark-brown liquid containing a high quantity of organic compounds and minerals generated by penetrating water through compacted waste layers [1–3]. The percolating rainwater through the waste brings about biodegradation and physicochemical reaction within solid waste thereby influencing the quantity and quality of the leachate to be generated. The quantity and quality of the leachate are also determined by the types of waste, the lifetime of the landfill, class of refuse, and its composition [4]. Its chemical complexity is directly related to a mixture of wastes variety [4–7].

Landfill leachate with various organic and inorganic constituents is in the type of highly polluted wastewater which shows acute and long-lasting toxicities [8–12]. When

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these contaminants are discharged into the water body, they can have a negative health effect on water biodiversity [13,14]. Because of continuous stringent regulations for treating leachate and its considerable costs, leachate treatment has become important in integrating and sustaining of municipal solid waste. The expenditure for the management of leachate generated by the solid waste industry is ranging between 750,000 and 14 million dollars. It is about (20%–33%) of operational expenses in landfills [15,16].

The first leachate management and control began in the 1970s [17–19]. The attempts started by focusing on the use of physical, biological, and chemical treatment processes that have been extensively applied in treating municipal wastewater. The treatment target was not achieved successfully because pollutants in leachate are usually more heterogeneous and refractory than wastewater. Most recently, many factors have been weighed in offering an ideal system of treatment that needs to have high pollution reduction performance. For the first time, advanced oxidation processes (AOPs) were used to treat biologically stabilized leachate in the 1990s [20–23].

AOPs are a set of oxidation processes in the aqueous phase which utilize efficient oxidizing agents such as hydroxyl radicals ($OH^{\bullet}$) and sulfate radicals ($SO_4^{\bullet-}$) [24–26]. They are considered among the powerful, environmentally-friendly, and comparatively low-cost techniques for treating water/wastewater [27–30]. They have been implemented broadly in treating wastewater due to their cost-effectivity and generating powerful oxidants that degrade refractory organic contaminants and eliminating some inorganic contaminants in wastewater that are resistant to conventional treatment methods [26,27,31–35].

At the time of proposing AOPs for treatment of water/wastewater in the 1980s until now, various oxidation methods examined and utilized. Chemical characteristics of pollutants and working conditions play a key role in determining the effectiveness of the treating techniques [27].

To treat leachate properly with the highest efficiency, the performance of various AOPs have been reviewed and summarized by Deng [36]. Chemical oxygen demand (COD) removal by ozonation was (6%–88%) with the mean value and a standard deviation (SD) of 53% and 24% respectively [36]. Furthermore, the efficiency for eliminating COD of the leachate by using ozone/H_2O_2 was in the range of 18% to 78%, while, its mean value and SD were 48% and 23%, respectively [27].

According to data obtained by Deng [36], it was found that 35% to 90% of COD was removed from leachate with the use of Fenton processes with the mean value and SD value of 71% and 13%, respectively. As a result of the bibliographical revision, Singh and Tang [37] found that the efficiency of the Fenton process to remove COD of leachate ranged from 31% to 95%. Fenton process is one of the most applied AOPs to degrade a wide range of recalcitrant organic compounds [34,38].

According to previous studies, there is no information on preferred pH when oxidation by ozone and Fenton process are used as hybrid techniques. The novelty of this research work is to find optimum conditions to enhance the removal efficiency of leachate pollution parameters by hybris AOP techniques.

2. Materials and methods

2.1. Sampling of landfill leachate

Raw leachate samples were collected on the dates stated here (September 20, 2018), (January 24, 2019) and (March 6, 2019) from Güngör sanitary landfill which is located in the north-east of Nicosia-Cyprus; its location and area are shown in Fig. 1a [39] and Fig. 1b respectively. The samples were collected in 0.5 L sterile sample bottles and to keep their natural characteristics, they were stored in the fridge at 4°C [40]. Characteristics of the raw landfill leachate are listed in Table 1 and it was observed that depending on the rainfall, the characteristics changed, as the collecting pool was open to the air.

2.2. Experimental procedure and design

Treatment of leachate with AOPs was divided into two classes. Single techniques: (ozonation, hydrogen peroxide, Fenton process) and hybrid techniques: (peroxone reaction (H_2O_2/O_3)) and (Fenton/O_3). Optimization of pH and contact time (CT) for O_3 treatment has been carried out in a batch reactor (1 L glass beaker) using OPAL OG 400 ozone generator with the flow-rate of 400 mg h^{-1}. The processes of H_2O_2 (30% Merck KGaA, Germany) optimization for "pH, dosage" and Fenton reaction for "pH, $[H_2O_2]$:$[Fe^{+2}]$ ratio and CT" were conducted under static conditions using (jar test VELP, FC 6S). The optimum pH and CT of the hybrid techniques have also been investigated. All AOPs in optimized conditions has been tested on the same leachate samples to compare their efficiencies based on their COD removal percentage (RP) and leachate pollution index (LPI RP). Fig. 2 illustrates methodological pathways for landfill leachate treatment with AOPs.

2.3. Leachate pollution index

LPI, which is a tool for quantifying the leachate pollution potential of landfill sites, was calculated based on the method provided by Kumar and Alappat [41]. Out of eighteen parameters utilized for calculating of LPI, nine parameters were available for analyses in the present study, as listed in Table 2. Hence, Eq. (1) which is a modified equation for calculating LPI used as described by Kumar and Alappat [41].

$$\mathrm{LPI} = \frac{\sum_{i=1}^{m} W_i P_i}{\sum_{i=1}^{m} W_i} \tag{1}$$

where LPI is the leachate pollution index; W_i is the weight of the ith pollutant variable, P_i is the subindex score of the ith leachate pollutant variable, m is the number of parameters utilized in counting LPI.

2.4. Analytical methods

The heavy metals in the raw leachate were determined using (Agilent 7500 Series ICP-MS system, USA), the biochemical oxygen demand (BOD_5) was determined by the

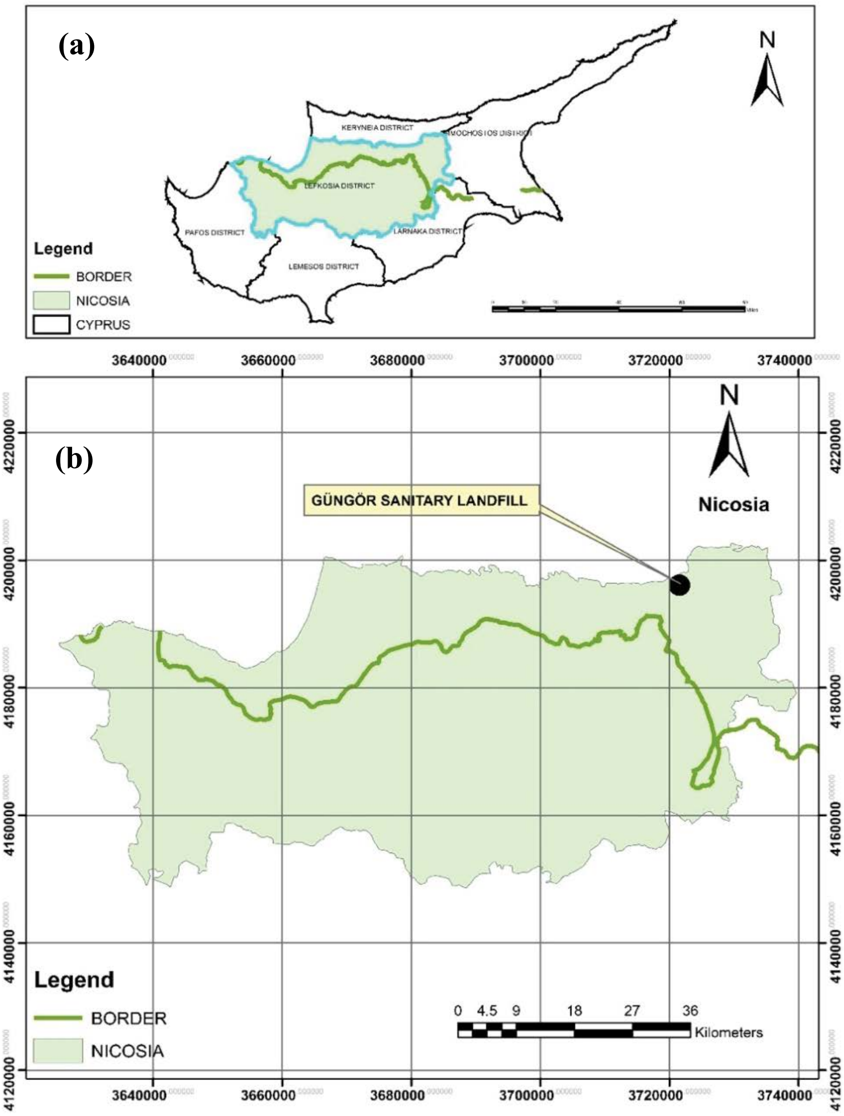

Fig. 1. Güngör sanitary landfill (a) location and (b) area [39].

respirometric method using OxiTop WTW kit (Germany), and COD was determined by the closed reflux, colorimetric method (Method 5220 C, Standard Methods).

3. Results and discussion

The initial COD of the leachate had some variance due to the sampling dates with different weather conditions. In this study, the optimum leachate treatment conditions in terms of COD RP by AOPs have been found. In the process of treating landfill leachate, the optimization has been done for O_3, H_2O_2, and Fenton process separately. Then, the singular optimized conditions were applied in hybrid techniques.

3.1. Ozone-based AOPs

The pH and CT optimization of landfill leachate in terms of COD RP by the ozonation process are shown in Fig. 3. The highest COD RP of 69.4%, 71.35%, and 81.23% was achieved at a pH of 4, 7, and 8.4 respectively under CT of 120 min. It is observed that ozone shows its best result in pH = 8.4 and CT = 120 min. Results with the ozonation process show that COD can be removed more effectively under basic pH. It is known that in ozonation under acidic pH, oxidation can be achieved only by molecular ozone, while in basic medium oxidation is dominantly carried out by less selective radical species (mainly $OH^{\bullet}$) [27,42,43]. Oxidation power is higher with hydroxyl radicals than with direct ozone molecule due to their higher oxidation potential that results in higher COD RP [44]. The formation of hydroxyl radical by O_3 has been proposed in various detailed methods and the overall reaction including $OH^{\bullet}$ formation is expressed in Eq. (2) [45].

$$3O_3 + H_2O \rightarrow 2OH^{\bullet} + 4O_2 \quad (2)$$

Table 1
Characteristics of the raw landfill leachate

Characteristics	Values
COD, mg L^{-1}	21,175
BOD_5, mg L^{-1}	1,650
App. color, Pt-Co	22,110
True color, Pt-Co	19,800
Turbidity, NTU	222
TDS, mg L^{-1}	24,960
EC at 25°C, μS cm^{-1}	41,311
Hardness, mg $CaCO_3$ L^{-1}	6,403
Total chromium (Cr), ppm	0.727
Nickel (Ni), ppm	0.717
Zinc (Zn), ppm	0.496
Copper (Cu), ppm	0.131
Selenium (Se), ppm	0.045
Cadmium (Cd), ppm	0.001
Antimony (Sb), ppm	0.022
Lead (Pb), ppm	0.015
Arsenic (As), ppm	0.205
Mercury (Hg), ppm	0.009

3.2. H_2O_2-based AOPs

The H_2O_2 dosage and pH optimization of landfill leachate in terms of COD RP by H_2O_2 are shown in Figs. 4a and b respectively. To find the optimal pH for the treatment of landfill leachate, 6 different pH have been tested. CT was kept constant at 120 min. Leachate treatment with H_2O_2 shows its best result (50.71% COD RP), in alkaline medium with pH 11 [46,47] and 4 g H_2O_2 L^{-1}. Increasing H_2O_2 dosage results in COD reduction until it reaches 4 g L^{-1}, it remained constant to 6 g L^{-1}, and thereafter by increasing H_2O_2, COD concentration starts to increase. This can be explained as follows: The COD test procedure depends on the simultaneous chemical reduction of Cr^{6+}, during the oxidation of the organic materials; keeping this process in mind, it is obvious that when the extra H_2O_2 is not used for organic materials degradation, it will be free in the medium and will lead to errors in the COD results because of the change of red-ox conditions. Further addition of H_2O_2 beyond its optimal dose, gives errors for COD readings. So, the increase can be misleading. That is why the optimum point is the endpoint of COD reduction.

Table 2
Weights of the pollutant parameters involved in LPI [41]

No.	Pollutants	Significance	Pollutant weight
1	BOD_5	3.902	0.061
2	COD	3.963	0.062
3	Copper (Cu)	3.170	0.050
4	Nickel (Ni)	3.321	0.052
5	Zinc (Zn)	3.585	0.056
6	Lead (Pb)	4.019	0.063
7	Total chromium (Cr)	4.057	0.064
8	Mercury (Hg)	3.923	0.062
9	Arsenic (As)	3.885	0.061

3.3. Fenton-based AOPs

The pH, $[H_2O_2]$:[Fe(II)] ratio, and CT optimization of landfill leachate in terms of COD RP by the Fenton process is shown in Figs. 5a–c, respectively. Leachate treatment with Fenton reaction shows its best result (88.68% COD RP) under a pH of 3.5, $[H_2O_2]$/[Fe(II)] ratio = 4:1.4 at 50 min CT.

3.3.1. Effect of pH on Fenton reaction

The typical pH range of the Fenton process that has been reported for the treatment of landfill leachate is 2.0–4.5 [37,48,49]. The importance of pH in effectiveness of the Fenton process in wastewater treatment has been confirmed by some scholars that include the stability of H_2O_2, iron speciation, and management of the oxidant and the substrate activities [50–52]. According to Sedlak and Andren [53], the rate of $OH^{\bullet}$ radical will be higher in the pH scale of 2.0–4.0 as a result of the organometallic complex reaction. This reaction will cause either the regeneration of $OH^{\bullet}$ or increasing reaction rate. Also, due to $OH^{\bullet}$ scavenging properties, eliminating inorganic carbon from wastewater will improve its treatment efficiency by AOPs [54] and it can be easily achieved in acidic conditions [55]. Fig. 5a shows the influence of pH on the leachate treatment by the Fenton process. The highest COD RP was achieved at pH 3.5 and based on that, subsequent experiments were conducted.

3.3.2. Effect of $[H_2O_2]$:[Fe(II)] ratio on Fenton reaction

The most two significant operational parameters in implementing the Fenton process are $[H_2O_2]$:[Fe(II)] ratio and organic matters to ferrous iron $[RH^{\bullet}]$:[Fe(II)]. Since both Fenton reagents are scavenging the hydroxyl radicals, molar ratio optimization of Fe(II) to hydrogen peroxide needs to be determined in the laboratories. Organic pollutants can be reduced in the Fenton process with both oxidation and coagulated iron sludge [56–60].

It needs to be considered that the elimination of particular organic pollutants is increased as the concentration of reagents rises, but at a certain threshold level, the treatment becomes negligible by increasing reagents dosage [48,52]. Both electrical conductivity (EC) and total dissolved solids (TDS) are highly iron dosage-dependent and increasing its amount in the effluent, add further treatment steps before sending it to discharging point [61]. Adding an excessive amount of H_2O_2 has a contribution in producing gas bubbles that result in sludge sedimentation inhibition [48,59] and may have negative effects on biological treatments [61].

To find the optimum $[H_2O_2]$:[Fe(II)] ratio, thirteen different ratios were tested with initial leachate COD strength of 12,980 mg L^{-1}. First, the hydrogen peroxide was used alone in a dosage of 4 g L^{-1} without adding ferrous iron, and then

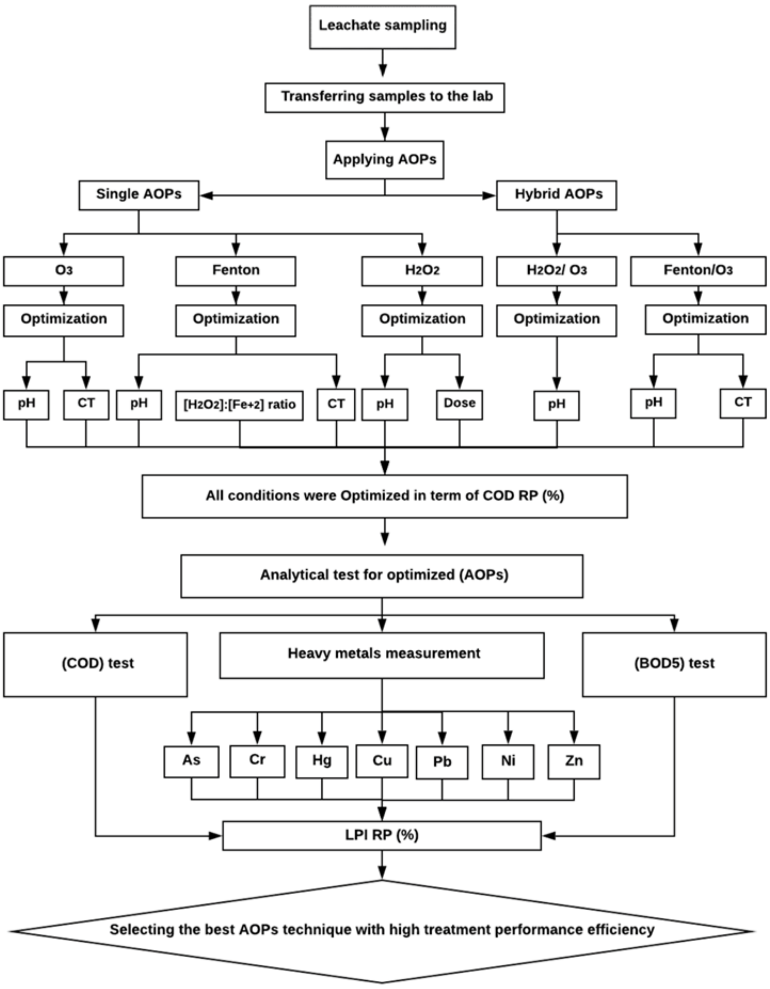

Fig. 2. Methodological pathway for landfill leachate treatment with AOPs.

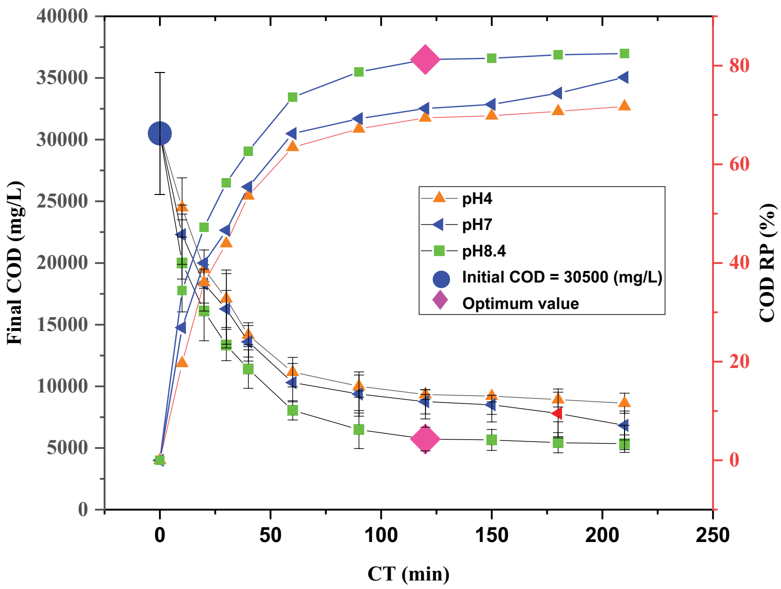

Fig. 3. Optimization of pH and CT in terms of COD RP for landfill leachate treatment using the ozonation process.

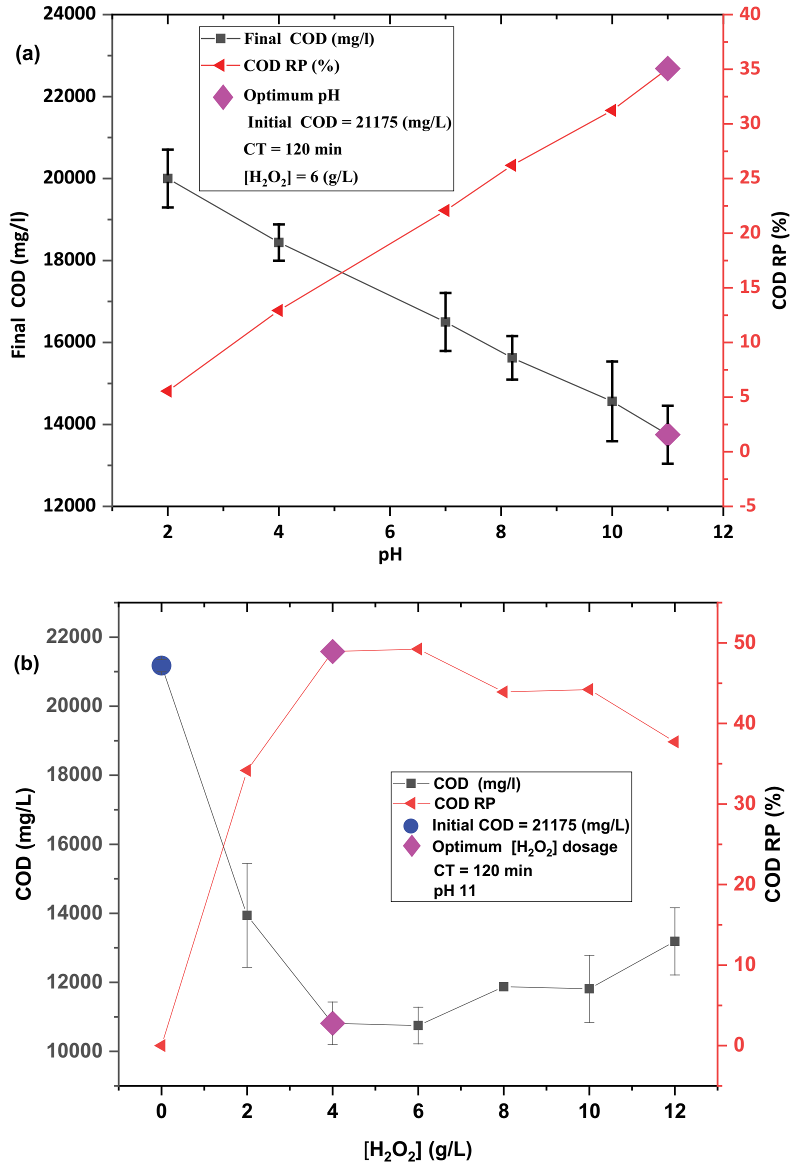

Fig. 4. Optimization of parameters in terms of COD RP for landfill leachate treatment using H_2O_2 (a) pH and (b) H_2O_2 dosage.

Table 3
LPI RP and COD RP of AOPs methods

Types	CT (min)	pH	Test no.	LPI	LPI RP (%)	COD RP (%)
Raw leachate	0	8.4	–	20.644	0	0
O_3	120	8.4	1	12.800	38.00	80.87
H_2O_2	120	11	2	15.937	22.80	50.70
Fenton process	50	3.5	3	9.190	55.48	87.54
H_2O_2/O_3	120	8.4	4	12.689	38.53	81.93
H_2O_2/O_3	120	11	5	11.642	43.61	86.54
Fenton/O_3	50	8.4	6	9.838	52.34	92.79
Fenton/O_3	120	8.4	7	9.548	53.75	93.79
Fenton/O_3	50	3.5	8	9.431	54.32	93.91
Fenton/O_3	120	3.5	9	8.906	56.86	95. 04

it was held constant in the same dosage while ferrous iron dosage changed from 0.2 to 2.4 g L^{-1}. Temperature, pH, and CT were kept at 20°C, 3.5, and 60 min respectively.

Fig. 5b shows the influence of $[H_2O_2]:[Fe^{2+}]$ ratio on the COD RP. Results are in good agreement with those reported by Zhang et al. [55]. According to Zhang et al. [55] in the treatment of landfill leachate with the Fenton process, the best result can be achieved in optimal $[H_2O_2]:[Fe^{2+}]$ ratio of 3. According to Haber and Weiss [62] report, the reaction becomes second-order and zero-order concerning the low and high concentrations of H_2O_2 and Fe^{2+} respectively. Hermosilla et al. [63] reported that there was no significant increase of COD RP in the hydrogen peroxide to the ferrous iron range of 5–7. It is related to the slow mineralization of highly recalcitrant organic materials [52] and the reducing ferric iron to ferrous iron with the generation of $OH^{\bullet}$ [63].

3.3.3. Effect of CT on Fenton reaction

The efficacy of Fenton processes in terms of COD RP has been also evaluated for its CT. Fig. 5c shows the increase in COD RP as a function of CT with the Fenton process at 20°C, pH 3.5, and $[H_2O_2]:[Fe^{2+}]$ ratio = 3. COD has been measured in 15 different CTs ranging from 3 to 327 min. It can be seen that the most organic and oxidizable compounds rapidly destroyed by H_2O_2 in the initial 50 min. With the prolonging of time, the COD RP decreased. The same situation was reported by Cetinkaya et al. [64]. It is thought that, when the refractory materials in leachate were oxidized, the residual

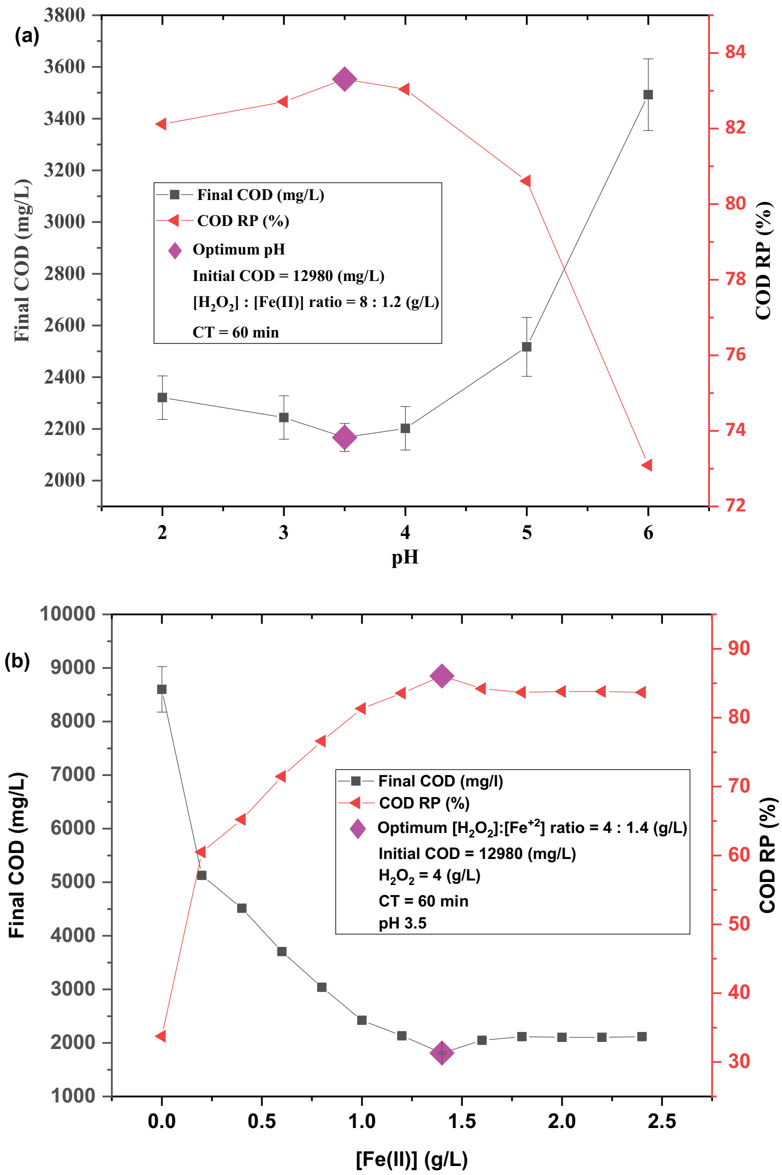

Fig. 5. Continued

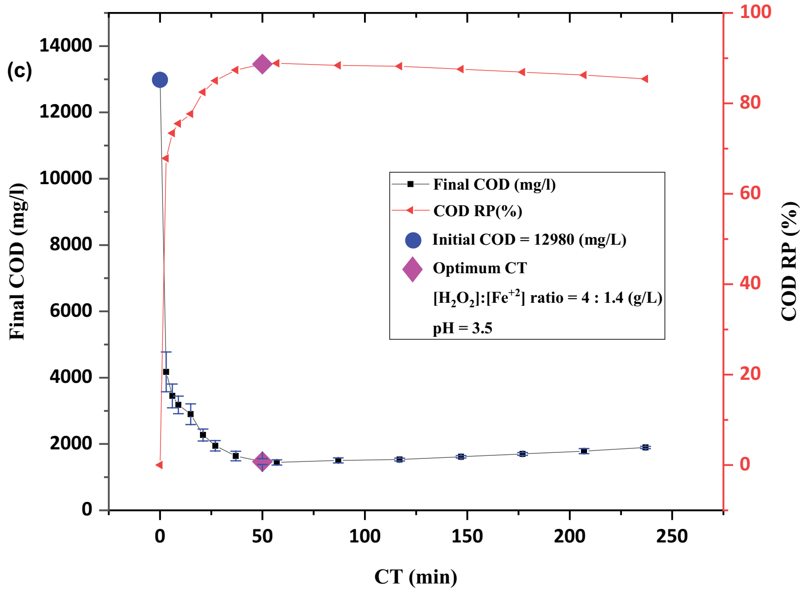

Fig. 5. Optimization of parameters in terms of COD RP for landfill leachate treatment using the Fenton process (a) pH, (b) $[H_2O_2]$:$[Fe^{+2}]$ ratio, and (c) CT.

materials were not easily degraded by $HO^{\bullet}$, so the efficiency became stabilized [65,66].

3.4. Efficiency comparison of single and hybrid AOPs in treating landfill leachate

After finding the optimum values for single AOPs including ozone, H_2O_2, and Fenton process, H_2O_2 and Fenton process was combined with ozone, to find the most efficient way to treat landfill leachate. Because they were showing optimum values in different pH and CT, hybrid methods of peroxone reaction and Fenton/O_3 tests have been applied in two and four different ways respectively to find their optimal conditions. Finally, LPI and COD RP have been calculated to choose the best and more efficient method in treating landfill leachate.

All kinds of single and hybrid AOPs techniques that have been tested in this research work are listed in Table 3 and their LPI RP and COD RP have been illustrated in Fig. 6. Based on the results, degradation percentage of contaminants by ozonation process improved by adding H_2O_2 which result in the generation of high potential and non-selective hydroxyl radical [67]. Compared to the Fenton process operations (Tests 3, 6, 7, 8, 9), ozone-based operations (1, 4, 5) were less effective.

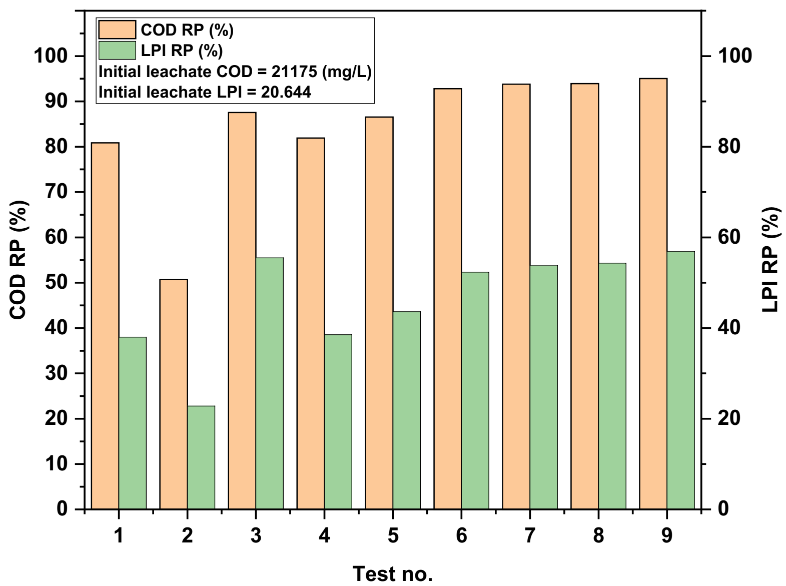

Fig. 6. Comparison of AOPs methods via LPI RP and COD RP.

Also, results show that Fenton/O_3 has the highest RPs for COD with LPI of the leachate in the acidic medium (pH = 3.5), but comparing to the use of the Fenton process alone, the reduction is not more significant. This is due to the low production of hydroxyl radicals at the acidic medium by O_3. Ozone did not show a significant contribution to the efficiency of the Fenton reaction process.

4. Conclusion

Based on the results found in this study, it was concluded that the COD removal of landfill leachate by H_2O_2 is a slow process in comparison to the use of H_2O_2/Fe^{2+} (Fenton process) or H_2O_2/O_3 (peroxone reaction). In the comparison of all tested AOPs, it has been found that using Fenton/O_3 as a hybrid technique in acidic medium with pH 3.5 after 120 min CT has the highest treatment efficiency. However, its superiority compared to the Fenton process is not significant. On the other hand, the Fenton process can be chosen as the best AOPs, economically. It is also concluded that the pH value of the treated media has a critical influence in determining the effectiveness of tested AOPs. Leachate treatment by the Fenton process at its optimum pH = 3.5, gives the best and most economical solution.

The LPI, as a well-accepted parameter in the leachate strength, was also reduced after the application of the above-mentioned processes (from 20.644 to 9.190). Further treatment of sanitary landfill leachate is possible only after its pre-treatment and reduction of conventional parameters to acceptable limits. LPI reduction ensures successful further treatment.

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