

original article

Catalytic oxidation of hydrogen peroxide and the adsorption combinatory process in leachate waste pretreatment from composting factory

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INTRODUCTION

The advanced oxidation process (AOP) and the wet air oxidation (WAO) process are regarded as the appropriate options for wastewater treatment, with moderate-to-high organic compounds.^[1] Catalytic wet air oxidation (CWAO), including oxidation of organic compounds in wastewater with CO₂, N₂, and H₂O at a temperature and pressure of

125 – 220°C and 5 – 50 bar, using oxygen or air as the oxidants in the presence of a catalyst, will be suitable.^[2] The WAO method had been first used for pre-treating industrial wastewaters with a high bar. In this process, high temperature, pressure, and air or oxygen injection are involved; moreover, pure metals, activated carbon, H₂O₂, and other oxidants are used to improve the process and increase the removal efficiency.^[3] The main limitation of this process is accumulation of a homogeneous catalyst H₂O₂, giving rise to an effluent pollution.^[2] Many efforts have been made to find heterogeneous catalysts having the appropriate catalytic activity. With regard to acidic conditions in a reaction environment, the iron and copper compounds that are fixed on activated carbon will be appropriate. Iron zeolites and various clay compounds are also used as catalysts in the CWPO process. The following mechanism can describe the catalytic activity:^[4]

ABSTRACT

Aims: The purpose of this study was to use a combinational process of catalytic oxidation of hydrogen peroxide (CWPO) and activated carbon for improving the removal efficiency of organic matter.

Materials and Methods: The effect of the operational parameters such as residence time (30 – 90 minutes), reaction temperature (100–300°C) in the pressure 10 bar, and catalytic iron concentration were investigated. The effect of the catalytic method, catalytic wet peroxide oxidation (CWPO), along with the absorption process, on granular activated carbon (GAC), powdered activated carbon (PAC), PAC/Cl₂, and GAC/Fe were considered; oxygen is used to supply pressure and H₂O₂ is applied as the main oxidant in various concentrations.

Results: The removal efficiency of the chemical oxygen demand (COD) was over 35%, obtained in one hour of retention time, with the wet air oxidation (WAO) process, and the removal efficiencies of GAC, PAC, and PAC/Cl₂, at a temperature of 300°C, and activated carbon concentration of 2 g/l, were 43.4, 38.9, and 33.6%, respectively.

Conclusion: These results indicate that the reaction temperature, residence time, and H₂O₂ dose are the most important factors affecting the degradation of organic matter. The GAC/Fe catalyst process had a higher efficiency than other absorbents for organic matter oxidation.

Key words: Composting factory, leachate, wet oxidation

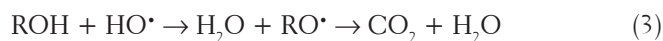
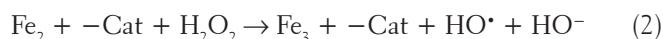
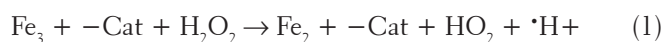
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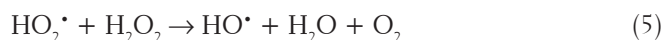
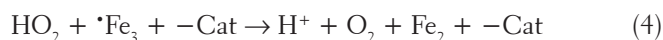


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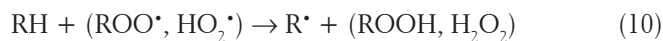
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The following side reactions are also carried out besides the main ones.^[5]



As there is a possibility of iron leaching in most cases; recently, a new catalyst, activated carbon-saturated free metal, has been used during the CWPO process. Due to hydrogen peroxide decomposing into oxygen and water instead of the production of hydroxyl radicals, on the activated carbon, the removal efficiency of these catalysts, to remove refractory pollutants, is low.^[4,5]



Rivas *et al.* studied the catalytic effect of metals and sulfate radicals on the wet oxidation of landfill leachate, and found that the removal efficacy of COD was reached in the range of 60 - 80% in the present of Oxone (sulfate radical).^[6] In another study, the combination of $\text{CuSO}_4 + \text{Na}_2\text{SO}_3 + \text{air}$ in the wet oxidation process increased the COD removal efficacy and biodegradability of the treated leachate.^[7] Li *et al.* investigated $\text{Co}\pm\text{Bi}$ and Mn/Ce complex oxides in the municipal landfill leachate treatment. The results indicate that Mn/Ce , with a higher specific area, had more COD removal efficacy.^[8] The hydrogen peroxide-promoted WAO with an Al/Fe catalyst had been employed by Galeano *et al.*, with an increasing catalyst concentration and a lower H_2O_2 dose, by which the removal efficiency of COD was improved. WAO was conducted at 200°C in the presence of Fe^{2+} . The COD removal efficiency of 90% was achieved in this case.^[9] In this study, the CWPO process was used with GAC/Fe catalyst. This catalyst was tested successfully by A. Quintanilla *et al.*, for phenol removal, in the CWAO process.^[5] In a study done by Suarez-Ojeda *et al.*, the CWAO catalytic process was also carried out using activated carbon (AC), to reduce the temperature and pressure, and control the intermediate compounds during the reaction with the aim of producing a wastewater with high degradation.^[4] The

main purpose of this study is to determine the COD removal efficiency from the Isfahan compost plant leachate, with the CWPO combinational process. It superficially adsorption through granular activated carbon (GAC), powdered activated carbon (PAC), PAC with calcium hypochlorite, and absorbed iron catalyst on the GAC also is investigated.

MATERIALS AND METHODS

The characteristic of leachate

The samples leachate were collected in 20 L polyethylene containers from the Isfahan compost plant and transferred to the laboratory. All the experiments were repeated five times at each stage, and the average and the removal efficiency were obtained. Some of the main physical and chemical properties of the raw leachate sample are described in detail in Table 1.

Setup of the wet peroxide oxidation process

The pilot includes a pressure autoclave made of (stainless) steel in a 3 L volume that can withstand pressures up to 100 bar and temperatures of 500°C . It is also equipped with a pressure drain valve, manometer, sample input and output, mixer, cooling system, and so on. The schematic of WPO is given in Figure 1.

Effect of different temperatures, contact time, and hydrogen peroxide concentration on wet peroxide oxidation efficiency

The study was done at temperatures of 100, 200, and 300°C , retention times of 30, 60, and 90 minutes, and at a pressure of 10 bar. To temperature adjustment, a heater, was used (HACH model), and to adjust the pressure and also for the supply of oxygen required for oxidation, a pure oxygen cylinder was applied. The 1, 2.5 and 5 ml of hydrogen peroxide was added to the reactor after sample. Before arriving sample into the reactor, preheating the reactor was conducted at temperature of 80°C for 2 h. The operational conditions are given in Table 2.

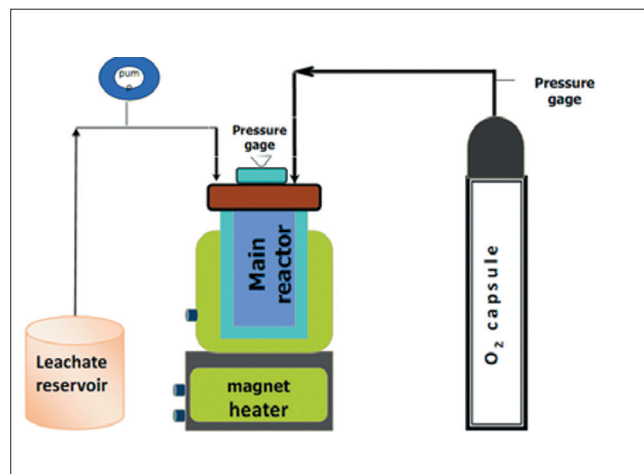


Figure 1: Wet peroxide oxidation reactor system

Table 1: Physical and chemical properties of the row leachate

Components	Concentration (g/L)		
	Mean	Maximum–Minimum	Standard deviation
COD	118.2	97.9–146.98	14.4
BOD ₅	75.19	54.5–99	15.3
Ammonium	180.2	105.8–360	99
Nitrate (mg/L)	578	500–680	67.2
TSS (mg/L)	3990	600–5800	2156
EC (µs/cm)	979	900–1100	62.8

Table 2: Wet oxidation operational conditions

Oxidizing agent	Pure oxygen and H ₂ O ₂
Partial Pressure of O ₂	10 bar
Temperature	100, 200, and 300°C
Volume of reactor and sample	3 L and sample volume 1500 cc
Duration of the reactions	1.5 hours after preheating period
Reaction time	30, 60, and 90 minutes
Cooling of reactor	2 – 3 hours
Mixing inside reactor	50 S ⁻¹

Effect of sorbent dose on removal efficiency

In order to improve the removal of organic matter, granular activated carbon (GAC), powdered activated carbon (PAC), and a combination of PAC and calcium hypochlorite (PAC/Cl₂) (with 70% active chlorine), and coated iron on granular activated carbon (GAC/Fe) were used. The quantities of 0.5, 1, 2, and 3 g/L of granular activated carbon were added to the reactor. For best results, pH solution was adjusted at 5.5, in accordance to the previous study.^[10] All types of activated carbon were purchased from Merck Company. Before placing the activated carbon (PAC and GAC) into the reactor, it was heated at a temperature of 105°C in a furnace for 24 hours, and then placed in the desiccator. All steps were taken and the preparation of granular activated carbon was made according to the study of Kihc *et al.*^[11] To make GAC/Fe, the GAC was added to the solution of ferric iron as FeSO₄ (2.5% weighted), at 3 pH, at 80°C, for 12 hours. After complete evaporation and iron adsorption, the activated carbon was heated at a temperature of 105°C in the furnace for 12 h and then dried in the desiccator. In order to remove the surface deposits of iron, the end product was washed twice with distilled water. Finally, the activated carbon was filtered (effective size of 0.5–1 mm) and dried at 100°C. All steps were taken and the preparation of GAC/Fe was made according to the study of Liou *et al.*^[12] For measuring the concentration of H₂O₂ in the filtered solution, the titration method with potassium permanganate was used. Thus, 250 ml of the sample was added to two erlenmeyer flasks; and 100 ml and 10 ml of H₂SO₄ was added. Next, the solutions were titrated by the standard of potassium permanganate 0.02 M, and the titration continued until the emergence of a pink color.^[13] After the reaction in the reactor and before measuring COD, the centrifuge was used at a speed of

6000 rpm for 15 minutes in order to separate the activated carbon from the output samples. Moreover, in order to remove the effect of H₂O₂ on the increase of COD, 0.1 of manganese oxide was added to the solution for 15 minutes before measuring COD.

Chemicals

The iron sulfate (FeSO₄·7H₂O), H₂O₂ (30% W/V), H₂SO₄, NaOH, Acetic acid (CH₃COOH), potassium dichromate (K₂Cr₂O₇), HgSO₄, Ag₂SO₄, manganese oxide, and powder and granular activated carbon were purchased from Merck.

Instrumentation

The COD was measured with the existing method in the Standard methods, Part D5220, APHA (1995).^[14] The amount of Fe coating on the Fe GAC surface was determined by a graphite furnace atomic absorption spectrophotometer (GFAA) (model 20AA Varian company), after being extracted by HNO₃ (10 wt %) solution for 12 hours.

RESULTS

The study was done to determine the effect of temperatures of 100, 200, and 300°C and retention time 30–90 minutes in various concentrations of hydrogen peroxide, along with the surface absorption process, with different kinds of activated carbons, GAC, PAC, PAC/Cl₂, and GAC/Fe.

The effect of temperature

To study the effect of temperature on the oxidation of organic matter, the experiments were performed in the absence and also presence of catalytic activated carbon and the results were drawn as the COD removal efficiency by four procedures of GAC, PAC, PAC/Cl₂, and GAC/Fe versus the temperatures of 100, 200, and 300°C. As seen in Figure 2, with increasing temperature, the COD removal efficiency also increased so GAC at a temperature of 300°C had 43.4% efficiency, and GAC/Fe with the maximum removal efficiency of 60%, showed the highest percentage of removal. The lowest removal efficiency was related to PAC/Cl₂, with 25%, at temperatures of 100°C and 300°C and a concentration of activated carbon of 2 g/l. The GAC removal efficiency was 43.4%, PAC, 38.9%, and PAC/Cl₂, 33.6%. Other results are given in Figure 2.

The effect of hydrogen peroxide concentration

The highest COD removal efficiency is observed in 90 minutes, in a concentration of 5 mg/l hydrogen peroxide, and a maximum efficiency of 39.4% is obtained under these conditions. The highest COD output concentration is also seen in the 30-minute retention time and 1 ml of hydrogen peroxide; in addition to this, increasing the retention time will increase the COD removal efficiency, as it is observed in Figure 3. In the hydrogen peroxide concentrations of 5 and 2.5 ppm, the COD removal efficiencies are close to each other. This can be due to the increase in organic acid resistance with low molecular weight, which leads to

decomposition in these two concentrations. Other results are given in Figure 3.

The effect of activated carbon concentration

Figure 4 indicates the effect of the CWPO process on COD concentration reduction. With an increase in the activated carbon concentration, the COD concentration decreases, so the average value of it reduces from 1133 g/l to 586 by 3 g/l of GAC. The average concentration of PAC and PAC/Cl₂ in 3 g of concentration was equal to 795 and 820 g/L, respectively, in which the concentrations of granular activated carbon are more. The other results are given in Figure 4.

The effect of granular activated carbon/Iron catalytic process:

Compared to GAC, the GAC/Fe catalytic process has a higher removal efficiency. In 3 g/l of activated carbon concentration, the COD removal efficiency is 48.2%, and it has reached 64.7% in the GAC/Fe catalytic process. According to Figure 5, the removal efficiency is increased by increasing the carbon concentration. Figure 5 shows

the values of COD adsorption by GAC, GAC/Fe, and PAC/Cl₂.

Kinetics of granular activated carbon/wet peroxide oxidation

The quantity of adsorbate will be affected by both the properties and concentration of the adsorbate and temperature. The most important characteristics of the adsorbate, which are effective in the absorption, are solubility rate, molecular structure, polarity, and saturation quantity of its hydrocarbons. In general, the quantity of adsorbate is determined as a function of concentration at a constant temperature, which is known as the absorption isotherm, and is determined by measuring the quantity of adsorbate in a fixed volume of activated carbon, in accordance with the following equation:

$$q_e = ((C_o - C_e) V)/m \tag{12}$$

where 'q_e' is the quantity of adsorbate (mg adsorbate/mg adsorbent), 'C_o', the initial concentration, 'C_e', the concentration after absorption, 'V', the volume of liquid in the reactor (1 l in this study), and 'm' the mass of activated carbon or adsorbent. Langmuir and Freundlich isotherms are often used for

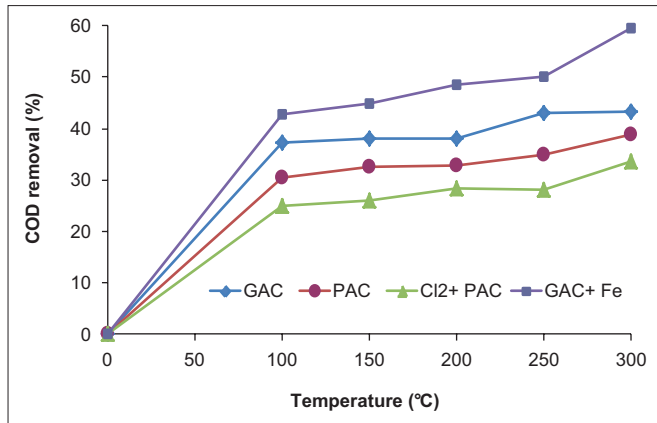


Figure 2: COD removal efficiency by four CWPO processes; GAC, PAC, PAC/Cl₂, and a combination of GAC/Fe, at different temperatures

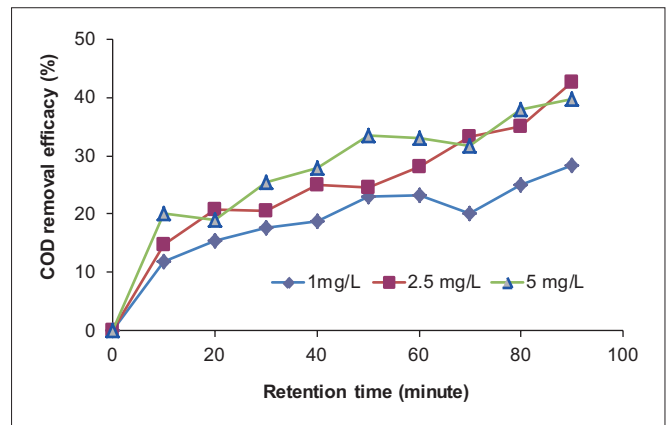


Figure 3: Effect of hydrogen peroxide on COD removal by catalytic wet oxidation at different times

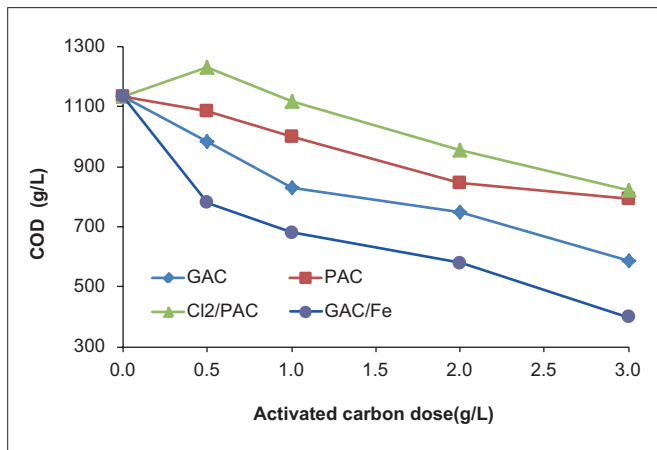


Figure 4: Amounts of COD concentration of the adsorption process by GAC, PAC, and a combination of PAC/Cl₂

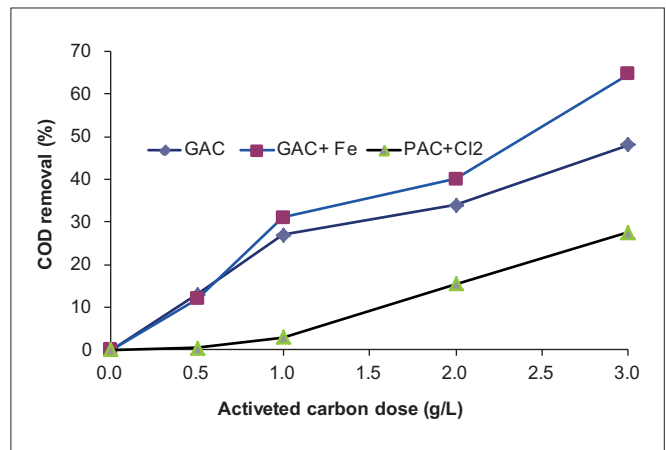


Figure 5: Adsorption values of COD by GAC, GAC/Fe, and PAC/Cl₂

absorption.^[15] The Freundlich Isotherm equation:

$$x/m = q_e = K_f C_e^{1/n} \quad (13)$$

'x/m' the adsorbate mass on the adsorbent after equilibrium (mg adsorbate/g activated carbon), 'K_f' the Freundlich factor (mg adsorbate/g activated carbon) × (L water/mg adsorbate) 1/n. The Langmuir and Freundlich isotherm results (GAC) are given in Table 3 and Figure 6.

In this study, an appropriate temperature of 200 – 300°C; retention time of 90 minutes, and optimal concentration of hydrogen peroxide 5 ml, were determined. The regression equation for COD (g/l) removal was obtained. The values of all the coefficients of Eq. 22 are given in Table 4.

DISCUSSION

Effect of different temperatures on wet peroxide oxidation efficiency

In general, an increase in temperature gives rise to an increase in COD removal efficiency, so COD removal efficiency increases with temperature; furthermore, the use of GAC has a higher efficiency than PAC and PAC/Cl₂. The GAC absorption capacity is better in this process. Temperature is a very important parameter for removing organic compounds in the CWAO process. Increasing temperature is followed by a combination of beneficial effects, which affect the reaction.^[16] According to the Arrhenius law, higher temperature of the reaction will lead to an increase in the reaction rate. Second, in temperatures higher than 100°C, oxygen solubility in water is increased greatly.^[17] However, from a practical point of view, it should be noted that higher reaction temperature will give rise to an increase in corrosion problems.^[18] Corrosion problems result from the by-products manufactured during the process, such as, carboxylic acids with low molecular weight that are formed during the reaction. Also, at higher temperatures, the costs of the process go up and control of the process becomes difficult.^[19] In this condition, the efficacy study of the catalyst becomes difficult; this is why the temperature should not usually exceed 300°C in the WAO process. In a study done by Liu *et al.*, with temperature increase, the COD and phenol efficiency are added. When the reaction temperature increases from 160 to 200°C, the COD and phenol efficiency is increased from 12.5 and 18%, respectively; however, when the CWAO catalytic process along with activated carbon resin are impregnated with Ru/KC, nearly 92% of COD and 96% of phenol removal efficiency has been obtained. With the temperature increase from 200 to 240°C, COD and phenol removal efficiency has increased very slightly.^[20] This phenomenon can be interpreted as being due to the increase in organic acids, with low molecular weight, which are more resistant to decomposition. Increasing these compounds at higher temperatures will lead to an rising in the resistance to oxidation.^[21]

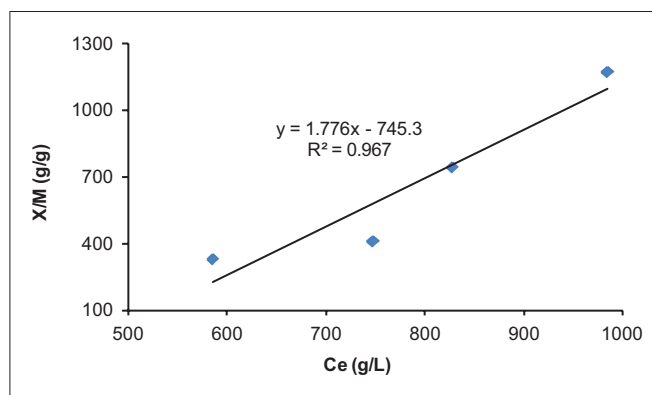


Figure 6: Langmuir isotherms for granular activated carbon

Table 3: Langmuir and Freundlich isotherms coefficient

C ₀	C _e	C ₀ -C _e	m	X/M	C _e /X/M
1570	1570	-	0	-	-
1570	985	585	0.5	994	0.990736
1570	828	742	1	717	1.154895
1570	748	822	2	576	1.29936
1570	586	984	3	290	2.023648

Table 4: Estimated regression coefficients for COD (g/l) removal

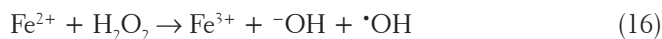
Term	Unstandardized coefficients		Standardized coefficients	t	Sig.
	B	Std. Error	Beta		
(Constant)	114.247	4.235		26.975	.000
Time	-.211	.047	-.346	-4.476	.000
Temperature	-.018	.008	-.168	-2.179	.031
H ₂ O ₂ dose	-4.866	1.411	-.266	-3.449	.001

Effect of contact time and hydrogen peroxide concentration on wet peroxide oxidation efficiency

Hydrogen peroxide (H₂O₂) has a high content of effective oxygen, low cost, safe storage method, and is very easy to use. Above all, it has no adverse effect on the environment. The standard value and potential reduction of hydrogen peroxide during the CWPO reaction are as follows:



The reaction is a strong indication of hydrogen peroxide being an acidic solution. Using catalysts, its oxidation power can be increased.^[22] In this study, to increase the effect of hydrogen peroxide, an iron catalyst was applied in the GAC combination, hence, the following reaction took place in the bed of activated carbon:



Thus, OH radicals were produced during the reaction. This reaction was capable of reducing the organic matter from the leachate.^[23] Experimental results has been showed that

WPO could effectively reduce the organic compounds in oil sludge to petroleum; retention time and reaction temperature were important factors for COD removal. At a reaction temperature of 340°C, the initial concentrations of oil sludge reached 4000 mg/l petroleum, in nine minutes retention time, and 88.7% of COD removal efficiency.^[24] The COD removal efficiency increased with an increase in the reaction temperature and retention time.^[25] The results of the study indicated that WPO could effectively remove the organic compounds from waste leachate. The COD removal efficiency could reach more than 44%. Reaction temperature, retention time, and hydrogen peroxide concentration were effective factors in organic matter decomposition.

Effect of sorbent dose on wet peroxide oxidation efficiency

The COD removal efficiency was improved by adding GAC to the reactor. The maximum removal efficiency for COD was 48% by this method, while the maximum WPO removal efficiency was 39.4%. Due to its synergist property, a combination of the WPO method with GAC activated carbon was considered an appropriate option for leachate treatment.^[26] Initial oxidation of organic compounds to a stable oxidized state, and also decomposition of coarse and resistant pollutants to small molecules were made by WPO; as a result, stable end products were formed like CO₂ and H₂O,^[18] formation of OH radicals, and also improvement of the surface absorption rate; it reacted rapidly with target compounds in the leachate through the OH radicals and resulted in the final decomposition of the leachate organic matter.^[27] Increased oxidation of organic compounds by WPO in the leachate depended on the initial value of GAC and reaction chain of WPO decomposition.^[15] In this case, by affecting molecule decomposition, WPO would lead to an absorption increase in the pyrrolic groups in a graphene layer in the GAC (which is the same electron basal level); thus causing a reduction in the micropore congestion in GAC, by which the absorption power would increase.^[28] The disadvantages of this method were, high consumption of energy and the need for revival of consumer GAC; in addition, the WPO method could be used for the recovery of activated carbon.^[29] Figure 4 indicates the Fe catalyst activity in the CWPO process, in various concentrations of the activated carbon, at a temperature of 300°C. As was observed, using this catalyst in higher concentrations of the activated carbon increased the COD removal efficiency. In a study conducted by Ning Li *et al.*, to remove chlorophenol by the catalytic process, with absorbed ruthenium on activated carbon, it was observed that Ru/ZrO₂ was very efficient at removing CP-2 during the CWAO catalytic process. Even the process efficiency was high (more than 90%) at low temperatures (393°K) and a total pressure of 3 MPa; moreover, CP-2 oxidation was added by increasing the temperature. The main parameters for removing chlorophenol included the molecular properties, presence of a benzene ring, and solubility rate.^[30]

Kinetics and isotherm study

In this study, the appropriate temperature 200 – 300°C; retention time 90 minutes, and optimal concentration of hydrogen peroxide, 5 ml, were determined. From the experimental adsorption studies it was seen that retention time, temperature, and the H₂O₂ dose were the most important factors affecting COD removal from the leachate.^[31] The regression equation for COD (g/l) removal is shown herewith. Values of all the coefficients of Eq. 17 are given in Table 4.

$$\text{COD (g/l)} = 114.247 - 0.211(\text{time}) - 0.018(\text{temperature}) - 4.866\text{H}_2\text{O}_2 \text{ dose} \quad (17)$$

With regard to drawing the graphs and creating a curve in the graph of the Langmuir isotherm, the use of the Freundlich isotherm is more appropriate; hence, considering the Langmuir graph, when X/M: 630, the quantity of C_e will be equal to 360 (the intersection with the y axis); and when X/M: 742, the quantity of C_e will be equal to 560; then K_f will be equal to 1.076, and the slope of the curve will also be equal to 1.776; thus, when C_e: 1, the equation will be X/M=1.0762 C_e.^[32]

CONCLUSION

The study considers the catalytic wet oxidation process with hydrogen peroxide along with various absorbents of GAC and PAC; and a combination of PAC/Cl₂ and also a fixed iron catalyst on GAC, to reduce the organic load from the compost plant leachate. The CWPO catalytic process along with GAC/Fe is very effective in oxidizing high concentrations of organic matter, to obtain 65% removal efficiency of COD. The COD consideration was strongly dependent on the remaining concentration of the oxidant (hydrogen peroxide). In general, the sequence of COD removal efficiency could be FeGAC/CWPO >> GAC/CWPO > PAC/CWPO > PAC/Cl₂/CWPO and WPO in five tested processes. The results in the laboratory scale showed that due to increased breakdown of the organic compounds into simpler ones at higher temperatures, using this method together with biological treatment (aerobic and anaerobic) could be a promising option for leachate treatment, as it caused a decrease in oxygen consumption in following processes.

It is necessary that other researchers make use of the results of this plan and apply this method on a semi-industrial scale and finally on a full scale. The problems of this study are precise adjustment of the pressure and temperature inside the reactor, the possibility of bursting pipes and fittings during an operation, preparation of FeGAC, the recovery of activated carbon, and so on. Based on the obtained laboratory results, it has been determined that the CWPO catalytic process has an appropriate efficiency for removing organic matter under optimal conditions, and this method can be used for strong sewage with a high organic load, like leachate of

compost plants or also a landfill, to reduce loads and increase biological degradation.

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