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Zeolite and Activated Carbon as Catalysts on Leachate Clarification

Banchón Carlos*

Environmental Engineering, Escuela Superior Politécnica Agropecuaria de Manabí Manuel Félix López, (ESPAM-MFL), Calceta 130602, Ecuador

Sigcha Pavlova, Gavilanes Paola

Faculty of Engineering and Applied Sciences, Environmental Engineering, Universidad de Las Américas (UDLA), Campus UDLAPARK, PB Vía Nayón S/N, Quito- Ecuador

Córdova Alexandra

Department of Chemical Processes, Faculty of Mathematical, Physical and Chemical Sciences, Universidad Técnica de Manabí (UTM), Portoviejo 130104, Ecuador
Postgraduate Coordination, Master in Hydraulics, Pontificia Universidad Católica del Ecuador (PUCE) – Manabí

*Corresponding author: carlos.banchon@espam.edu.ec

Improper solid waste management worldwide has increased the negative impacts of landfills due to the production of methane, carbon dioxide, and leachate wastewater. In the present work, granular activated carbon (GAC), zeolite (Ze), and hydrogen peroxide were used for the purification of landfill leachate. Emphasis was given to decreasing operational costs for a big-scale advanced oxidation process. Thus, the aim was to evaluate the effect of oxidant and catalysts dosages, and different highly basic pHs. Up to 95% of dark brown colour and 100% of turbidity from landfill wastewater were removed. Based on the experimental findings, it is suggested that an application of activated carbon and hydrogen peroxide in a dosage ratio between 1.7 and 2.0 would be economically attractive in terms of reduced operation costs.

Keywords: landfill leachate, Fenton's oxidation, coagulation, SARS-CoV-2.

Introduction

Terrestrial and aquatic species have suffered a level of extinction due to anthropogenic effects. Up to 84% of freshwater wildlife has become extinct since 1970

due to water pollution (Lau et al., 2020; UN Water, 2020; Jones et al., 2021). A rapid increase in plastic pollution is being found in water resources, soils, and

sediments due to 33% of worldwide municipal solid waste (MSW) which is not being managed in an environmentally safe manner (Munir et al., 2021; The World Bank, 2021). By the year 2025, it is expected that municipal solid waste generated per person per day will increase from 0.11 to 4.54 kilograms by 4.3 billion urban residents (Muthu, 2021; Show et al., 2019). Regions like Asia, the Pacific, and Europe generate around 45% of the world's total MSW, landfills being the first choice of waste management (Munir et al., 2021). Nevertheless, landfill operation is related to anthropogenic greenhouse gas emissions, contamination of surface and groundwater by pungent odours, bioaerosols, and wastewater leachate (Abiriga et al., 2020; Njoku et al., 2019). The wastewater as a product of rainwater percolation through solid wastes in landfills is defined as leachate. High concentration of organics like humic acids, hydrocarbons, pharmaceuticals, hormones, antibiotics, microplastics, and inorganics like heavy metals, most of them refractory and toxic, are the main substances of leachate; this is why leachate contamination from landfills raises major public concerns (Smaoui et al., 2018; Miao et al., 2019; Cheng et al., 2021). For example, dissolved organic matter concentrations in some leachates ranged from 800 to 70 000 mg/L or higher (Kamaruddin et al., 2017; Teng et al., 2021). Even recent research has already confirmed viral particles of SARS-CoV-2 in wastewater and primary sewage sludge in several countries. Viral survival in the environment in landfill leachate is still unknown (Anand et al., 2022; Kitajima et al., 2020; Kweiner et al., 2020; Li et al., 2021). Little is documented about the performance of concentration, extraction, and detection methods for SARS-CoV-2 in landfill wastewater (Anand et al., 2022). Overall, the discharge of landfill wastewater leachate into the environment causes detrimental effects on the aquatic life, infertility of soil, and mutagenic effects on humans (Asaithambi et al., 2020).

The physicochemical and biological characteristics of leachates depend on their age. Leachates are young (less than 5 years), intermediate (5–10 years), and old (more than 10 years) (Tałałaj et al., 2021). For young landfill leachates, the BOD/COD ratio is approximately

greater than 0.5, but for old leachates, the BOD/COD ratio is approximately less than 0.1 (Chen et al., 2021). For young leachates, conventional biological treatment (aerobic, anoxic, or anaerobic) is employed due to its high biodegradability (Teng et al., 2021). However, intermediate and old leachates require chemical treatment as their recalcitrant content is high (Cirik and Gocer, 2020). Notably, the BOD/COD ratio decreases with time; therefore, old leachates are more difficult to treat biologically (Kamboj et al., 2020). Yet, there is variability in composition and strength, and the proper design of leachate treatment is challenging. In general, physical-chemical, and biological treatments are the main methods to treat landfill leachate (Costa et al., 2019). The physical-chemical treatments include coagulation-flocculation, absorption, oxidation, and membrane separation, while biological treatments are used to remove organics and nitrogen as they are cost-effective in such removal (Miao et al., 2019; Teng et al., 2021). Indeed, the biological process might be inhibited by heavy metals like lead, nickel, chromium, silver, cadmium, barium, and mercury (Wijekoon et al., 2022). Despite this, diverse technologies have been developed at the lab scale with important results: adsorption, chemical precipitation, electrocoagulation, ultrasonication, electrochemical advanced oxidation processes like electrochemical oxidation, electro-Fenton, and sono-electro-Fenton processes (Aziz et al., 2007; Zhang et al., 2012; Asaithambi et al., 2020; Kundariya et al., 2021; Dereli et al., 2021; Hussain et al., 2022).

Advanced oxidation processes (AOP) combine hydrogen peroxide as an oxidant and iron as a catalyst to decompose recalcitrant contaminants. Furthermore, it is widely accepted that AOP is performed under acidic conditions. However, because oxidation methods require large amounts of chemical oxidants in a narrow acidic pH range, industrial applications of this technology for wastewater are limited (Teng et al., 2021). In contrast, there is evidence of up to 80% organic degradation with granular activated carbon (GAC) as a catalyst and H_2O_2 at pH levels greater than 8 (Kurniawan and Lo, 2009; Boczkaj and Fernandes, 2017). Catalysis is enhanced by the GAC's large surface area, pore size dispersion, polarity, and hydrophobicity

(Khalil et al., 2001; Rivera-Utrilla et al., 2002; Iwanow et al., 2020). In addition, zeolites (Ze) have higher thermostability, little or no corrosion, no waste or disposal issues, good selectivity, acid strength, and are easier to set up for continuous processes than conventional catalysts (Perot and Guisnet, 1990). In this work, GAC and Ze are employed as catalysts due to their physicochemical advantages over other materials. This study's novelty and significance are dependent on the fact that it was conducted in settings that were not acidic. GAC, Ze, and hydrogen peroxide were employed to purify landfill leachate at basic pH, hence preventing an acidic condition that would affect the operational costs of a large-scale AOP.

Materials and Methods

Sampling

Leachate wastewater samples were obtained from a landfill pond in Chimbo, Ecuador (*Fig. 1*). This is a highland city (2448 m.a.s.l.) with a population of 15 000. The age of the landfill leachate is less than 1 year. Samples were transported at 4°C to the laboratory. All the procedures for chemical oxygen demand (COD), oxidation-reduction potential (ORP), turbidity, conductivity, pH, and colour were referred to Standard Methods for the Examinations of Water and Wastewater (American Public Health Association, APHA, 2017).

Catalysts activation

Two catalysts obtained from local markets were studied independently: Zeolite (Ze) Ca-clinoptilolite type (average Si/Al: 4.35) and granular activated carbon (GAC) both with a sieve size 0.85 mm N° 20 (ASTM E11-87).

The activation of the catalysts was performed as follows: (i) screening and washing of the materials to remove dust; (ii) physical activation by high temperature; and (iii) chemical activation by a strong acid.

After washing and screening to remove all dust, 100 grams of GAC and 100 grams of Ze were heated at 515°C for 3 hours. Thereafter, 5 M nitric acid (Merck, USA) was added to each material in a proportion of

Fig. 1. Sampling of leachate wastewater from a landfill pond



6 mL per gram. This acidic mix was maintained for 12 hours to allow chemical activation. After that, pH neutralization was done with demineralized water. Finally, the catalysts were dried at 100°C.

Coagulation-flocculation

Based on earlier research, a coagulation-flocculation step was performed prior to the AOP process to increase the removal of suspended particles (Banchon et al., 2022). As coagulant and flocculant, 3000 ppm ferric chloride (99%, Fengbai, China) and 30-ppm anionic high molecular weight polymer polyacrylamide (Henan, China) were applied, respectively. In a jar test (Velp, Italy), the chemicals were mixed at 250 rpm for 2 minutes. After mixing, the entire volume was allowed to rest for 30 minutes without stirring.

Advanced oxidation process

Following the addition of FeCl_3 , a residual iron content remained in the system. A NaOH solution (Merck, USA) was then added to enhance coagulation. The aim was to improve the removal of suspended particles through the formation of iron hydroxide. In light of this, AOP above pH 7 was conducted in the present study. *Table 1* shows GAC and Ze treatments ranging from 4000 to 20 000 mg/L in an effort to reduce catalyst use in comparison to earlier research. 4000 to 12 000 mg/L of hydrogen peroxide (Merck, USA) was added with or without the presence of both catalysts. The initial pH of leachate was 8.5. To increase turbidity removal, the pH of the leachate was changed to between 9.0 and 9.5.

According to *Table 1*, leachate from earlier coagulation-flocculation was treated with varying concentrations of H_2O_2 and catalysts in a 700 mL beaker with 250 rpm mixing in a jar test (Velp, Italy) under UV-light. After 4 hours, the supernatant was collected for measurement of turbidity, colour, and COD.

Table 1. Oxidation process for both catalysts zeolite (Ze) and granular activated carbon (GAC)

Treatments	pH	Catalyst dosage (mg/L)	Hydrogen peroxide (mg/L)	Catalyst/Oxidant
T1 - GAC / Ze	8.5	0	12 000	0.0
T2 - GAC / Ze	8.5	4000	4000	1.0
T3 - GAC / Ze	8.5	10 000	8000	1.3
T4 - GAC / Ze	8.5	20 000	12 000	1.7
T5 - GAC / Ze	9.0	0	12 000	0.0
T6 - GAC / Ze	9.0	4000	4000	1.0
T7 - GAC / Ze	9.0	10 000	8000	1.3
T8 - GAC / Ze	9.0	20 000	12 000	1.7
T9 - GAC / Ze	9.5	0	12 000	0.0
T10 - GAC / Ze	9.5	4000	4000	1.0
T11 - GAC / Ze	9.5	10 000	8000	1.3
T12 - GAC / Ze	9.5	20 000	12 000	1.7

To calculate the contaminants removal, the following equation was computed:

$$\% \text{ Removal} = \frac{\text{Co} - \text{Cf}}{\text{Co}} \quad (1)$$

Where: Co – initial contaminant concentration;
Cf – final contaminant concentration.

Optimization

Tests were optimized based on initial attempts conducted in *Table 2* studies. Different concentration of H_2O_2 ranging from 6600 to 40 000 mg/L were applied while maintaining a catalyst concentration of 20 000 mg/L. The more effective catalyst between GAC and Ze was chosen for this optimization experiment.

Table 2. Optimization experiments

Treatment	Catalyst dosage (mg/L)	Hydrogen peroxide (mg/L)	Catalyst/Oxidant
O1	20 000	6600	3.0
O2	20 000	8000	2.5
O3	20 000	10 000	2.0
O4	20 000	13 300	1.5
O5	20 000	20 000	1.0
O6	20 000	40 000	0.5

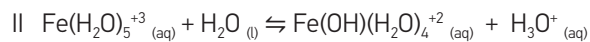
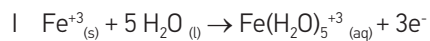
Results and Discussion

Effect of coagulant

The application of FeCl_3 as coagulant was previously evaluated in removing 90% of turbidity and 95% of COD (Banchon et al., 2022). The addition of FeCl_3 had two consequences: (i) suspended solids removal in the first stage, and (ii) a Fenton reaction under the presence of H_2O_2 and GAC/Ze. In other works, a suspended solids removal up to 95% was observed at 1509 ppm using FeCl_3 at pH 7.02 (Moradi and Ghanbari, 2014); and colour removal at pH 4, 6 and 12 (Aziz et al., 2007).

Coagulation neutralizes negative electrostatic charges from leachate colloids (Aziz et al., 2007); thus,

coagulation reduces repulsive forces among negative charged particles by the production of protons. When an iron salt is dissolved in water, it loses 3 electrons from its last orbitals, according to reaction (I). Consequently, the hydration of the ion occurs, and it takes the form $\text{Fe}(\text{H}_2\text{O})_5^{+3}$. This is because the iron atom in its electronic configuration still has 5 electrons to share. In reaction (II), the bond between the positively charged metal ion and an oxygen atom of one of the five water molecules in the $\text{Fe}(\text{H}_2\text{O})_5^{+3}$ results in a complex ion behaving like a proton donor due to the increased polarity of the O-H bonds of the water molecule. Therefore, hydrogen atoms have a greater tendency to ionize. This hydrolysis of the metal cation and proton production make the solution acidic.



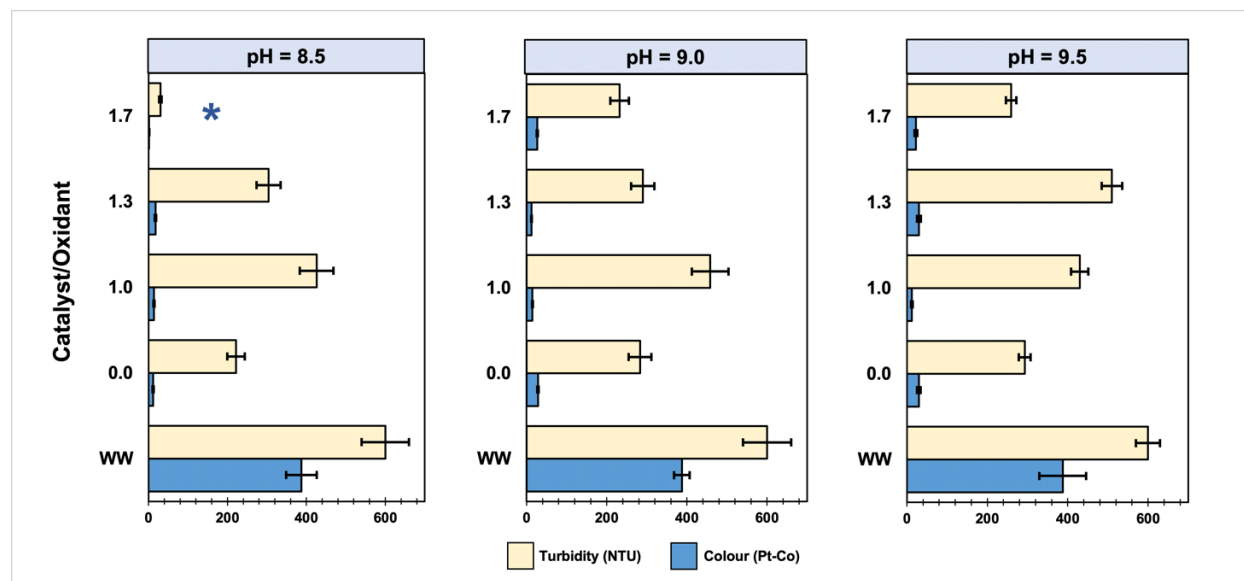
Effect of GAC

One of the most influential parameters in AOP is pH because it affects the catalysis of H_2O_2 on the carbon surface and its decomposition rate. Therefore, Fig. 2 shows the effects of basic pH (8.5, 9.0 and 9.5) and different H_2O_2 and GAC concentrations on turbidity and colour removal. In the present study, the colour

of landfill leachate changed from dark brown to transparency in treatment using a GAC/ H_2O_2 of 1.7 at pH 8.5. The colour changed from dark brown to light orange at pH 9.0 and 9.5 when using GAC. Turbidity and colour removal efficiency decreased at higher basic pH of 9.0 and 9.5. The dark colour of leachate samples indicated the presence of humic compounds, which constituted for the majority of organic components (Smaoui et al., 2018). In the present work, pH above 7.0 was considered because basic pH is a characteristic in young, intermediate, and old landfill leachate.

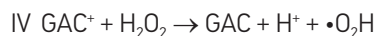
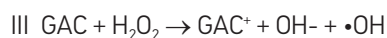
At pH 9.0 and 9.5, turbidity removal ranged from 92% and 97%, while colour removal from 15% to 61%. Higher GAC/ H_2O_2 dosages at higher pH did not improve the colour removal. At pH 8.5, the oxidant without GAC reached a 97% of turbidity removal, but a 63% of colour removal. In all treatments, H_2O_2 as oxidant without GAC was not capable to remove colour efficiently. At pH 8.5, the contaminants removal increased with increasing GAC dosages. The best treatment was a GAC/ H_2O_2 ratio of 1.7 to remove 95% of colour and 100% of turbidity. In other studies, a GAC/ H_2O_2 ratio of 3.8 at a pH 8.0 was used to remove 80% of chemical oxygen demand (COD) (Kurniawan and Lo, 2009). The same pattern was observed to removed up to 60% of organic matter using GAC/ H_2O_2 at pH 8.0 (Fan et al., 2007).

Fig. 2. Effect of initial pH on turbidity (NTU) and colour (Pt-Co) removal when using GAC as catalyst

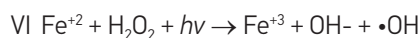
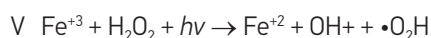


Acidic medium is reported as favourable when using H_2O_2 because the oxidation potential ($E^\circ = 1.77$ V) increases with the decrease of the pH (Lücking et al., 1998; Teranishi et al., 2016). Normally, under acidic conditions, there is a generation of hydroxyl radicals ($HO\cdot$) with a higher oxidation potential ($E^\circ = 2.8$ V), which is higher than chlorine, permanganate, and even ozone (Wang and Xu, 2012). Nevertheless, according to literature, an acidic environment was not effective for the removal of organic compounds from leachate (Kurniawan and Lo, 2009). In leachate treatment, it was observed that H_2O_2 decomposition was favourable at basic pH to produce $HO\cdot$ and OH^- (Boczkaj and Fernandes, 2017).

A hypothesis for the mechanism of oxidation is based on the porous structure of GAC which promotes the formation of $\cdot OH$. However, a reaction mechanism using activated carbon as a catalyst is not yet entirely researched. Reactions (III) to (VI) show that H_2O_2 decomposes catalytically by the presence of GAC (Khalil et al., 2001; Kurniawan and Lo, 2009). Heterogenous decomposition of H_2O_2 on activated carbon is considered to depend on both carbon porosity and the chemical properties of the surface (Khalil et al., 2001; Lücking et al., 1998).



The effect of residual Fe^{+3} ions from previous coagulation is also important to promote a Fenton reaction. In case of H_2O_2 and cations like Fe^{+3} and Fe^{+2} , the H_2O_2 decomposes to produce hydroxyl radicals ($\cdot OH$). These radicals are highly reactive species that can aggressively degrade organic matter (Wang et al., 2018). Besides, remaining Fe^{+3} ions from the coagulation stage contribute to the $\cdot OH$ formation as a Fenton reaction according to reactions (V) and (VI):



Effect of Ze

Fig. 3 depicts the effects of pH on the removal of turbidity and colour while applying Ze as a catalyst. As with the prior treatment, Ze/ H_2O_2 effectively removed suspended solids but not colour. In other investigations,

initial pH under UV-A irradiation affected the removal efficiency of persistent pollutants, resulting in high mineralization via Ze/ H_2O_2 (Perisic et al., 2016).

According to Fig. 3, at pH 8.5, 1.0 Ze/ H_2O_2 removed 99% of turbidity and 44% of colour. At 1.3 Ze/ H_2O_2 , 96% of turbidity and 50% of colour were removed. Zeolite in a Ze/ H_2O_2 ratio of 1.7 removed 86% of colour and 93% of turbidity.

At pH 9.0, 1.0 Ze/ H_2O_2 removed 98% of turbidity and 55% of colour. Ze/ H_2O_2 ratio of 1.3 removed 99% of turbidity and 66% of colour. Zeolite reduced 53% of colour and 98% of turbidity at a catalyst/oxidant ratio of 1.7.

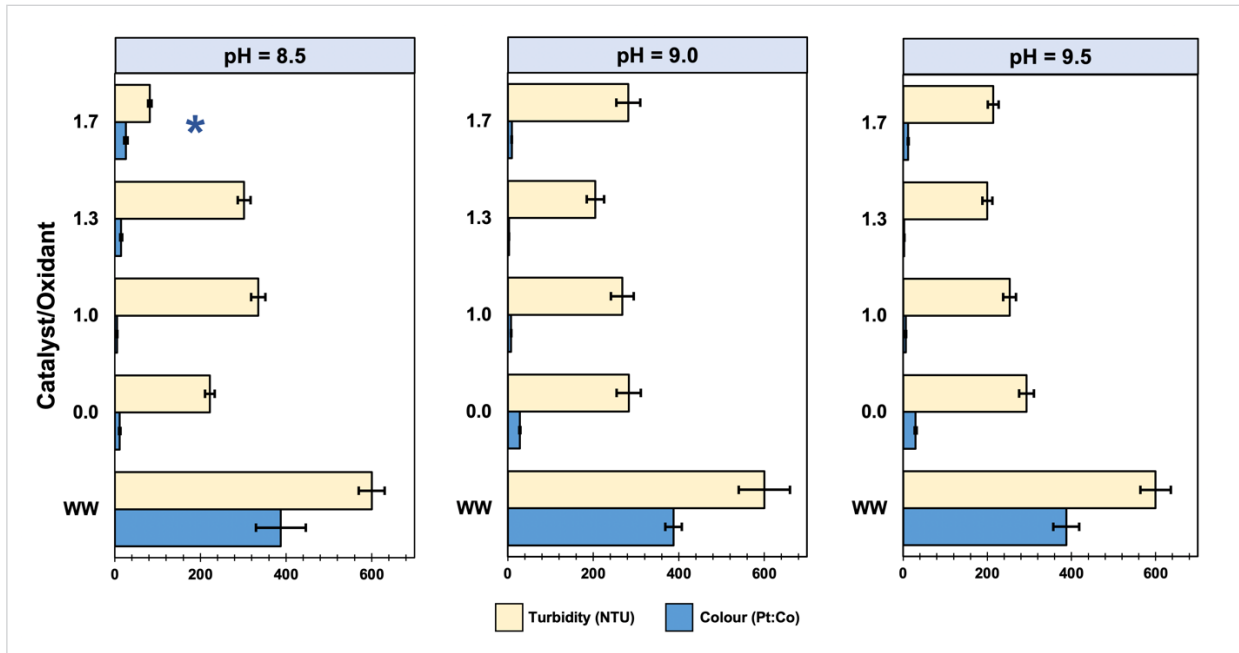
At pH 9.5, a Ze/ H_2O_2 ratio of 1.0 removed 98% of turbidity and 58% of colour. At a Ze/ H_2O_2 ratio of 1.3, a 99% removal of turbidity and 67% of colour were obtained; finally, zeolite in a catalyst/oxidant ratio of 1.7 obtained a 64% of colour and 97% of turbidity removal.

Evidence has been provided to consider that H_2O_2 alone is not effective to degrade organics (Chin et al., 2009). According to Figs. 2 and 3, at a Ze/ H_2O_2 ratio of 0.0, when dosing H_2O_2 oxidant alone, there was no significant removal efficiency. It turns out that H_2O_2 is a weak acid with relatively high oxidation potential but without $HO\cdot$ generation. An increase of H_2O_2 concentration led to an adverse effect on contaminants removal because a scavenging effect on the electrophilic $HO\cdot$ radical (Boczkaj and Fernandes, 2017). As shown in the results, there was no scavenging effect from H_2O_2 because Ze was added in excess. In contrast, a low catalyst/oxidant ratio showed good performance at basic pH in comparison with the results from Kurniawan and Lo (2009). In the present work, when dosing chemicals at pH 8.5, an efficient removal of colour and turbidity was obtained, although it is generally accepted that acidic conditions are preferred for AOP. As catalyst, Ze showed a good adsorption pattern to degrade suspended solids and 86% of colour particles at pH 8.5 (Fig. 3). In other works, Ze addition at an oxidation process enhanced decomposition of ozone into $\cdot OH$ (Buthiyappan et al., 2016; Ranga et al., 2016).

Optimization

Previously, a ratio of 1.7 catalyst/oxidant resulted in a greater catalyst efficiency for either GAC or Ze treatments. Yet, GAC effectively removed colour.

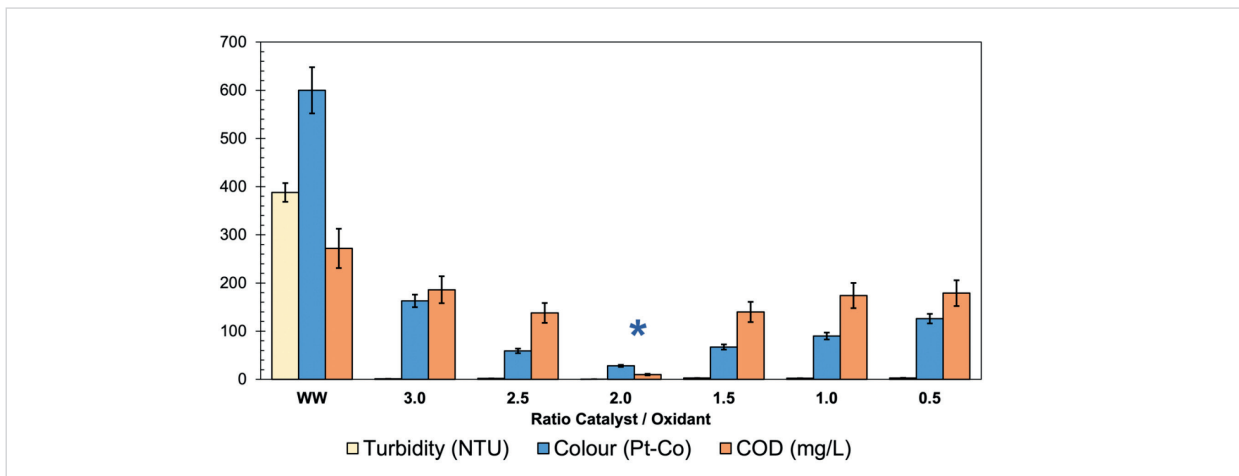
Fig. 3. Effect of initial pH on turbidity (NTU) and colour (Pt-Co) removal when using Ze as catalyst



In Fig. 4, additional AOP tests were conducted to determine the appropriate concentration of GAC/H₂O₂ at pH 8.5. Significantly, the 2.0 GAC/H₂O₂ ratio allowed for maximum reduction of organic materials. The final COD measurement is well below the maximum contamination level (200 mg/L) set by environmental authorities. At ratios between 1.5 and 2.0, up to 100%

of the turbidity was effectively removed. With GAC/H₂O₂ ratios between 1.7 and 2.5, up to 95% of the colour was removed. Other investigations corroborate, in accordance with the present findings, that organics in terms of COD were optimally degraded up to 96% when the GAC/H₂O₂ ratio was between 1.5 and 2.0 (Fan et al., 2007; Kurniawan and Lo, 2009).

Fig. 4. Effects of GAC/H₂O₂ ratios on turbidity (NTU, colour (Pt-Co) and COD (mg/L) removal



Conclusions

GAC, Ze, and H_2O_2 concentrations were studied at a range of pH levels to determine how they affected the efficacy of the AOP treatment in removing colour and turbidity. The current investigation demonstrated positive benefits to remove colour, turbidity, and chemical oxygen demand at a basic pH, contrary to the generally held belief that AOP is done under acidic conditions. At a pH of 8.5, the colour and turbidity were both eliminated by 86% when zeolite was used in a Ze/ H_2O_2 ratio of 1.7, and by 95% and 100%, respectively, when GAC/ H_2O_2 was used in the same concentration. Results show that catalysis is improved by decomposing H_2O_2 with GAC at a basic pH. When suspended particles were removed using

iron chlorine as a coagulant prior to the AOP, the Fenton reaction was boosted by the presence of hydrogen peroxide thanks to the existence of residual iron ions. The results of the experiments presented here imply that a combination of activated carbon and hydrogen peroxide applied at a basic pH could significantly lower operating expenses. Therefore, in order to stabilize costs, further research in large-scale operations is essential.

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