Performance of Phenolic Wastewater Degradation with Ozonation and Catalytic Ozonation Technique in Multi Injection Bubble Column Reactor

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Performance of Phenolic Wastewater Degradation with Ozonation and Catalytic Ozonation Technique in Multi Injection Bubble Column Reactor

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Abs 17.1. This research was developed for the degradation of phenolic compounds, specifically for phenol (C6H5OH) and 2,4-Dichlorophenol (2,4-DCP) as one of the most toxic phenolic compounds and widely used as a basis for pesticid 26 or herbicides and the like in the agricultural world. These phenolic compounds have a huge risk and potential danger to the environment and human health. Therefore, the processing of industrial wastewater contaminated with phenolic compounds is very important for environment 25 rotection. In this study, the ozonation technique was focused on accelerating the decomposition process through the formation of hydroxyl radicals (• OH) by increasing the initial pH of the solution (pH 12), which maximized the degradation of phenolic wastewater. Ferolite, an iron-oxide-based compound (FeO/Fe₂O₃), which is impregnated into active sand, is used as a catalyst mainly due to its good adsor 22 n ability, good catalytic effectiveness, and high availability. This experimental study was carried out for 60 minutes in a semi-continuous multi-injection bubble column reactor. Optimization of the degradation process in this case is obtained by observing various variable effects such as wastewater flow rate and air flow rate for ozonation, as well as the amount of catalyst for catalytic ozonation. The optimum conditions for the treatment process obtained by non-catalytic ozonation process in multi-injection bubble column reactors can be effectively used for phenolic wastewater treatment reaching 99.48% for phenol degradation and 99.83% for 2,4-DCP degradation.

INTRODUCTION

Phenolic compounds are one of the most common micropolytes in many industrial wastes such as petrochemicals, pharmaceuticals, steel industries, and the textile industry [4]. Phenolic compounds include a broad spectrum of organic compounds and their derivatives, with phenol, chlorophenol, and amino phenols being the most common [5]. Phenol has high chemical oxygen (COD) requirements with low biodegradability. Bey have harmful effects on living organisms, and further harm human health even at low concentrations [3]. Exposure to phenolic compounds, especially in one of the most toxic chlorophenol compounds in general, namely 2,4-dichlorophenol (2,4-DCP) which can cause damage to the kidneys, liver and pancreas, weakens the central nervous system, and denatures protein molecules. Because of this and its refractive properties, they have been included in the priority list of US 3 vironmental Protection Agency (EPA) pollutants, and consider phenols and their derivatives to be the 11th out of 126 chemicals in the class of high-risk pollutants [2]. Phenology 31st as most organic compounds find their way into the environment by removing from industrial wastewater. The Environmental Protection Agency (EPA) has set a phenologonic matter and the protection of the protection of drinking water sources by industrial activities. In drinking water, the maximum phenol concentration is $0.5 \mu g/L$ and for 2.4-DCP it is $0.03 \mu g/L$.

Various methods have been used so far to eliminate excessive amounts of phenols and phenol compounds including using biological processes, chemical oxidation, solvent extraction, combustion, rev oscillation methods [6]. However, most of these methods have disadvantages such as high cost, low efficiency, long time, and produce excessive by-products [1]. Here, the most effective method for rer 28 ng organic pollutants is the use of ozonation at high pH, which is considered an advanced oxidation process (AOP), because of the rapid decomposition of ozone in water, producing hydroxyl radicals with high oxidation potential values. Some of the advantages of this method, including ease of use, low cost, and high efficiency in addition to non-hazardous organic products. The opportunity to accelerate the ozonation reaction is to use a heterogeneous or homogeneous catalyst. Some metal oxides and ions have been studied and sometimes significant accelerations in the decomposition of target compounds have been achieved, although the mechanism of reaction in various cases is not vet clear.

This study investigated phenol and 2.4-DCP degradation techniques using ozonation and catalytic ozonation with iron oxide (FeO/Fe₂O₃) based catalyst in a multi-injection bubble column reactor. This study also studied the effect of ozone generation temperature on phenol and 2.4-DCP degradation. The multi-injection system produces a more homogeneous system with greater mass transfer and contact area so that oxidation occurs quickly and intensively in the bubble column reactor. It is expected that ozonation and catalytic ozonation in this study have a good performance in degrading phenolic wastewater. Spectrophotometric analysis with UV absorbance was used to measure the level of decomposition in each process, while the amount of COD concentration was used as a comparison of biodegradability during the degradation process.

MATERIALS AND METHOD

Materials

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Pure Phenol and 2,4-DCP compounds were obtained 111n Sigma-Aldrich. Ferrolite, granular active sand is obtained as a water treatment package and is ready for use. All solutions are prepared using demineralized water. In addition, all chemicals used in this work have extra purity or analytical level.

Methods

The experimental instruments or equipment used were semi-continuous bubble column reactors with specifications of 12 L, 190 cm and 9 cm respectively in volume, height and diameter. The reactor is equipped with a multi-injection system to supply more homogeneous ozone into waste water. The equipment is used to evenly distribute ozone from 3 dielectric release ozonators into w15 water. Ferrolytes are placed in a 200 micron stainless steel wire bag tied to stainless steel wire above the reactor. Fig. 1 shows a schematic diagram of the experimental setup for the ozonation technique.

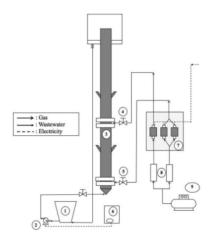


FIGURE 1. The schematic diagram of experimental set-up for ozonation technique

TABLE 1. Description of schematic diagram

1	Reservoir	6	Power Supply
2	Pump	7	Ozonator Circuit
3	Bubble Column Reactor	8	Flowmeter
4	Gas Injection 1 (I ₁)	9	Compressor
5	Gas Injection 2 (I ₂)		

The range of variations that will be evaluated in this study is determined by the hydrodynamic test, which consists of wastewater streams (255, 405, 495 mL / min) and air flow (8, 10, 12 L / min). Furthermore, they were investigated to obtain optimal conditions, which were then applied in a Ferrolite-based catalytic ozonation process, whose performance was evaluated by varying the amount of catalyst in weight (50, 100, and 200 g). The two subsequent experiments were carried out at room temperature (29 °C) by maintaining the initial pH (12) of wastewater. The process was then carried out for 60 minutes, treated samples were collected and analyzed every 15 minutes of reaction time, then degradation efficiency was recorded.

All samples were reacted with Merck Millipore phenol test kit 1.00856.0001 and the concentration of phenolic compounds was measured by spectrophotometer at $\lambda = 512$ nm. The degradation efficiency was calculated via Equation (1), where C_t is the concentration of phenol or 2,4-DCP after several regular intervals of time (t) and $C_{t=0}$ is the initial concentration.

Degradation efficiency (%) =
$$\frac{c_{t=0} - c_t}{c_{t=0}} \times 100$$
 (1)

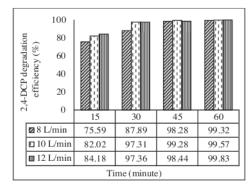
RESULTS AND DISCUSSION

Phenolic Wastewater Degradation by Ozonation

The Influence of Air Flow Rate on Phenol and 2,4-DCP Degradation

In this chapter, the air flowrates of 8, 10, and 12 L/min were investigated to obtain the optimal condition of ozonation degradation process. The multi-injection system were used to inject the ozone gas through the middle column (I_1) and the lower column (I_2) of the reactor from ozonators to the wastewater with volume of 15 L. The initial concentration of phenolic solution was prepared to be 50 ppm corresponded with some operating conditions

including waste water flow rate of 0.495 L/min, initial pH of wastewater (pH 12), and degradation time for 60 minutes at room temperature (29°C). The effect of air flow rates on degradation efficiency were served in Fig. 2(a) and Fig. 2(b).



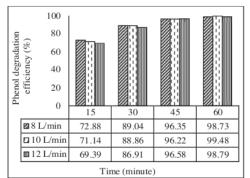


FIGURE 2. Effect of air flow rates of (a) 2,4-DCP, (b) phenol degradation efficiency ($C_{t=0} = \pm 50$ ppm, $Q_w = 0.495$ L/min, V = 15 L, $pH_0 = 12$, $\sum Q_{g,2,4-DCP} = 12$ L/min, $\sum Q_{g,phenol} = 10$ L/min)

The air flow rate range correlates with the amount of oxygen passed through the ozonator to produce ozone; whereas an increase in air flow rate affects more amount of oxygen available to produce more ozone gas, so that more hydroxyl radicals are formed. In addition, high air flow rates will provide a broad gas-liquid contact area that offers few benefit for the degradation process. This condition explains the performance of 12 air flow rate in Fig. 2 (a) above, that the highest degradation efficiency of 2,4-DCP reaches 99. 1% at a high air flow rate of 12 L/min with the remaining concentration of 0.085 ppm. Therefore, the condition of the 12 L/min air flow rate is then used as the control variable for the next part of the 2,4-DCP degradation process.

As observed from each time interval of Fig. 2(a), the inct 6 se of degradation efficiency was highest in the first 15 minutes and slows down over time. This correlates to the collision theory, in which the rate of reaction depends on the reactant particles colliding w 6 energy in excess of the Activation Energy for the reaction. As the degradation of 2,4-DCP progresses, 2,4-DCP are being turned into products so the number of 2,4-DCP particles left is decreasing. Consequently, the chance of 1 cessful collisions also decreases. For the next part of the 2,4-DCP degradation process, the condition of the 12 L/min air flow rate is then used as the control variable.

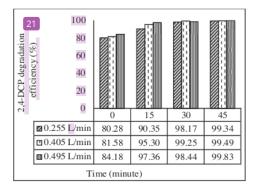
Based on Fig 2(b), the effect of the airflow rate on the phenol degradation efficiency increases very rapidly in the first 30 minutes of process, while the results after this range of minutes (in 45 minutes and 60 minutes) experience an increase which are not as significant as the precedent time. This phenomena could happen because of the hydroxyl radicals formed in the first 30 minutes were concentrated dominantly to attack phenol compounds in the wastewater. The hydroxyl radicals are expected to crack all chemical bonds of phenol compounds until it started to form other organic compounds as the intermediates derived in the process which leads to the complete degradation in the wastewater into CO₂ and H₂O. These organic compounds could be formed as quinone compounds, carboxylic acids, long-chain alcohol compounds, ketone/aldehyde compounds, and peroxide compounds. After undergoing a 30-minute process, the presence of intermediate compounds in the wastewater interfere the hydroxyl radicals attack on phenol compounds, so that some concentration of hydroxyl radicals become reacted with other organic compounds which resulting in decreasing rate of phenol degradation efficiency. Even so, the influence of airflow rate on phenol degradation percentage keeps showing uplift results over time until it reach 99.48% degradation efficiency.

However, a high air flow rate can also have a negative impact which leads to an increase in the fluid flow rate and further decreases the residence time. Based on the performance reputed in Fig. 2 (b), the air flow rate of 10 L/min has optimal conditions for phenol degradation compared to the air flow rate of 8 L/min and 12 L/min. It occurs because of the moderate air flow rate (10 L/min) which has the potential to provide a sufficient gas-liquid contact area with an adequate residence period. 12 s condition effectively degrades phenol to 99.48%, leaving a concentration of 0.26 ppm. The condition of the air flow rate of 10 L/min was then selected to be used as control variable for the next section for phenol degradation. In comparison to phenol, the increase of air flow rate for 2,4-

DCP degradation does not have a negative impact to the degradation efficiency. The highest air flow rate of 12 L/min effectively degrades 2,4-DCP until 99.83%. This shows that the effect of ozone and hydroxyl radical concentration is more prominent than residence time for the degradation of 2,4-DCP.

The Influence of Wastewater Flow Rate on Phenol and 2,4-DCP Degradation

The effect of waste water flow rates (0.255, 0.405, and 0.495 L/min) on degradation efficiency of ozonation was evaluated in this chapter with optimum air flow rate extracted from previous section. The operating conditions of this evaluation consist of initial concentration of phenolic solution was 50 ppm, initial pH of wastewater (pH 12), and degradation time for 60 minutes at room temperature (29°C) . The result was then presented in Fig. 3(a) and Fig. 3(b).



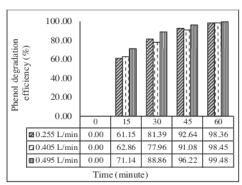


FIGURE 3. Effect of wastewater flow rate of (a)2,4-DCP, (b) phenol degradation ($C_{t=0} = \pm 50$ ppm, $Q_{g,2,4-DCP} = 12$ L/min, $Q_{g,phenol} = 10$ L/min, V = 15 L, $pH_0 = 12$, $\sum Q_w = 0.495$ L/min)

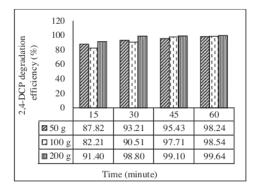
Wastewater or circulation rate contributes to the residence time of liquid in the reactor, which affected the contact time between the liquid and ozone [7]. The higher wastewater flow rates would give an effect to the duration of residence, which the residence time was longest at 0.255 L/min and shortest at 0.495 mL/min. The longer residence time would provide longer contact time between wastewater and ozone. However, the system equipped in this study is semi-continuous and a portion of the liquid remains in the reservoir. This condition expected the circulation of liquid from the reservoir to the liquid remains in the reservoir. This condition expected the circulation of liquid from the reservoir to the liquid remains in order to increase the degradation of liquid and increased the degradation rate, hence it resulted the highest degradation efficiency of 99.48% and 99.83% for phenol and 2,4-DCP system, respectively. Therefore, 0.495 L/min is determined as the optimal condition for wastewater flow rate to be used in further sections for phenol and/or 2,4-DCP degradation process.

The rate of phenol degradation efficiency as the effect of wastewater flow rate from Fig 3(b) has similar trend to phenol degradation efficiency rate of air flow rate in the previous section, which shows a rapid increase in the first 30 minutes. As stated before, the reason behind this phenomena still sticks to the concentration of hydroxyl radicals behavior which previously attack on phenol compounds dominantly in the first stage of degradation process (30-minute process), so that the value of phenol degradation rate shows a high result and the concentration of this compound becomes very quick to be reduced or degraded. When other organic compounds (intermediate compounds) are formed in the processed wastewater after 30-minute, the hydroxyl radicals tend to react with all compound that makes the rate of phenol degradation getting slower, yet still reach 99.48% degradation efficiency as its optimum condition.

Phenolic Wastewater Degradation by Catalytic Ozonation

In this experiment, the influence of the amount of catalyst used was evaluated with optimized variables on a single ozonation, at 10 L/min (for phenol degradation), 12 L/min (for 2,4-DCP) for air flow rate and 0.495 L/min for wastewater flow rate condition. The operating conditions consist of initial concentration of phenolic solution was 50

ppm, initial pH of wastewater (pH 12), and degradation time for 60 minutes at room temperature (29°C). Variation in the mass of the catalyst was set to be 50 g, 100 g, and 200 g Fe-based catalyst an d the results were illustrated in Fig. 4(a) and Fig. 4(b) below.



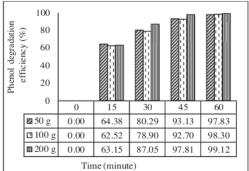


FIGURE 4. Effect of mass of catalyst of (a)2,4-DCP, (b) phenol degradation ($C_{t=0} = \pm 50$ ppm, $Q_{g,2,4-DCP} = 12$ L/min, $Q_{g,phenol} = 10$ L/min, $Q_{w} = 0.495$ L/min, V = 15 L, $pH_0 = 12$, $\sum m = 200$ g catalyst)

Fig. 4 above generally indicates that degradation efficiency was enhanced with increase of reaction time and catalyst mass. Based on Fig. 4(a), the highest degradation efficiency was obtained at catalyst load of 200 g as the optimum condition, which reached 91.40%, followed by catalyst load 100 g and 50 g, which reached 87.94 and 82.04% respectively. This result was consistent until the end of the reaction time, where the 200 g catalyst load achieved the highest degradation efficiency of 99.64% after 60-minute process. Based on Fig. 4(b), 50 g catalyst load achieved degradation efficiency of up to 97.83% in 60 minutes, which is approximately the same amount degraded (97.81%) using 200 g of catalyst after 45 minutes. Therefore, this shows that the latter (200 g catalyst) is a bit faster than the other, which then postulates that an increase in the amount of catalyst results in a more efficiency of catalytic ozonation was finally lower than that of ozonation, although the difference between the two methods was not significant. This is predicted due to the saturation of the catalyst over time are that the presence of wire mesh bag in which the catalyst was placed in increases pressure drop and decreases the contact surface between the gas phase and liquid phase. Therefore, it can be noted from this study that the presence FeO and/or Fe₂O₃ in ferrolite aids the initial degradation efficiency of phenol of phenol and 2,4-DCP but has a negative impact for longer reaction time.

Method's Result Comparison

TABLE 2. Method's result comparison

Method	Phenol Degradation	2,4-DCP Degradation
	(%)	(%)
Ozonation	99.48	99.83
Catalytic	99.12	99.64
Ozonation		

From the results, 2,4-DCP is generally easier to degrade than phenol because of the substitution of hydrogen by two electron donors (from the Cl functional group 5 which activate the aromatic ring. As a result, in this position 2,4-DCP reacts more actively with ozone, with the initial attack of the ozone notice which occurs in the ortho position or the hydroxyl group or negatively charged group of oxygen 3 ecause there is a chlorine atom bound to the position of the hydroxyl group or a negatively charged oxygen group 2,4-DCP, the addition of electrophilic from the ozone molecule to the para molecule to its position results in the release of functional groups or chlorine atoms.

From the observations obtained, both for phenol and 2,4-DCP have the degradation efficiency results for the ozonation technique that are slig 16 higher than the catalytic technique. In the ozonation method, at initial pH 12, ozone tends to break down into hydroxyl radicals ($^{\bullet}$ OH) which are capable of degrading organic pollutants, due to their high oxidative characteristics. The presence of Fe in catalytic techniques could cause the production of many intermediate radicals, including, HO₂•, $^{\bullet}$ O₂-, H₂O₂, etc., which have oxidation potential smaller than $^{\bullet}$ OH, thus limiting the process. However, the rate of degradation of this method increases to a certain extent with a high amount of Fe catalyst, whose advantages will be to compete and over-consume O₂• radicals that form in place, leading to the cessation of chain reactions for hydroxyl radical production [8], leading to negative effects on oxidation of organic matter.

CONCLUSIONS

In general, it can be concluded that the results obtained with lower degradation efficiency due to the presence of FeO and / or Fe2O3 in ferrolytes. The degradation 13 ciency of the single process reached 99.48% for phenol and 99.83% for 2,4-DCP, leaving a final concentration of 0.26 mg/L and 0.085 mg/L. Further results revealed optimum conditions achieved at moderate degrees of air movement (10 L/min) for phenols and high degrees of air movement (12 L/min) for 2,4-DCP, and waste water flow rates (0,495 mL/min) at initial pH = 12. This excellent condition was subsequently applied in the catalytic ozonation process, using 200 g of F13 hich resulted in a degradation efficiency of 99.12% for phenol and 99.64% for 2,4-DCP with a final concentration of 0, 44 mg/L and 0.19 mg/L.

ACKNOWLEDGEMENTS

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