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Ratnawati<sup>1a)</sup>, Enjarlis<sup>1b)</sup>, Slamet<sup>2c)</sup>

<sup>1</sup>Department of Chemical Engineering, Institut Teknologi Indonesia, Tangerang Selatan, 15320, Indonesia, <sup>2</sup>Department of Chemical Engineering, Faculty of Engineering, Universitas Indonesia, Depok 16424, Indonesia,

a)Corresponding author email: rnwt63@yahoo.co.id

b)en\_jarlis@yahoo.com
c)slamet@che.ui.ac.id

Abstract. The chemical oxygen demand (COD) and phenol removal from pharmaceutical wastewater were investigated using configuration of two circulation batch reactors in a series with ozonation and photocatalytic processes. The ozonation is conducted with O<sub>3</sub>/granulated activated carbon (O<sub>3</sub>/GAC), whereas photocatalysis with TiO<sub>2</sub> that immobilized on pumice stone (PS-TiO<sub>2</sub>). The effect of circulation flow rate (10; 12; 15 L/min) and the amount PS-TiO<sub>2</sub> (200 g, 250 g, 300 g) were examined. Wastewater of 20 L was circulated pass through the pipe that injected with O3 by the ozone generator, and subsequently flow through two GAC columns, and finally, go through photoreactor that contains photocatalyst PS-TiO2 which equipped with mercury lamp as a photon source. At a time interval, COD and phenol concentration were measured to assess the performance of the process. FESEM imaging confirmed that TiO2 was successfully impregnated on PS, as corroborated by EDX spectra. Meanwhile, degradation process indicated that the combined ozonation and photocatalytic processes (O<sub>3</sub>/GAC-TiO<sub>2</sub>) is more efficient compared to the ozonation and photocatalysis alone. For combination process with the circulation flow rate of 10 L/min and 300 g of PS-TiO2, the influent COD of around 1000 ppm are effectively degraded to a final effluent COD of 290 ppm (71% removal) and initial phenol concentration of 4.75 ppm down to 0 ppm for 4 h which this condition fulfill the discharge standards quality. Therefore, this portable prototype reactor is effective that can be used in the pharmaceutical wastewater treatment. For the future, this process condition will be developed for orientation on the industrial applications (portable equipment) since pharmaceutical industries produce wastewater relatively in the small amount.

#### INTRODUCTION

Nowadays, the environmental problems are faced by countries in the word. The pharmaceutical industry is one of the most significant sectors in the world's economy especially in the countries that have big population. The increasing demand of medicine gives the positive impact on economic. On the other hand, it also creates the negative impact on environmental problems. High concentration pharmaceutical industry wastewater needs the high degree of treatment before it discharged into the environment [1]. Since it categorized as harmful wastewater, conventional treatment such as physical, chemical, and biological methods are not adequate to treat this waste [2]. Recently, Advanced Oxidation Processes (AOPs) such as ozonation [3,4] and photocatalysis [5] are wastewater treatments that provide the high removal of pollutants. In ozone process, the availability of O<sub>3</sub> and OH radicals that have the high oxidative power play an important role in the degradation of pollutants. For instance, high efficiency of ozone process for drinking water system has been studied [6], however, ozone process is relatively expensive due to the consumption of electricity. Meanwhile, photocatalysis with semiconductor TiO<sub>2</sub> is a green technology for degrading wide variety organic pollutants with solar energy [4,5], and other environmental application such as air quality control, CO2 removal and disinfectant of bacteria [7,8]. Moreover, semiconductor TiO<sub>2</sub> is an effective photocatalyst due to the thermal stability, low toxicity, noncorrosive and inexpensive [9]. Photocatalysis with TiO2 impregnated on pumice provides some advantages since pumice can act as adsorbent and the capability of pumice to float improves photon absorption. In addition, TiO2 impregnated on the pumice also reduce the difficulty in the filtration of the photocatalyst since filtration can be eliminated by immobilizing TiO<sub>2</sub> on the solid support such as pumice [5,10]. However, photocatalysis still has limitation since its low oxidation rate, only suitable for the dilute wastewater and in some cases the complete mineralization is slowly [4]. In general, a combination of several treatment methods provides the high efficiency removal of pollutants compared with individual method [11]. Therefore, the combination of ozone process and photocatalysis is reasonable for treatment pharmaceutical wastewater that indicated the high concentration of pollutants. This process is required in order to fulfill the discharge standards quality in short time treatment process. Actually, some researchers have investigated this combination, however, they used synthetic and single waste such as formic acids, aniline, pyridine, humic acid etc [4]. To the best of our knowledge, the application of this combination has not been investigated yet for the real wastewater especially pharmaceutical wastewater that consists of hardly degradable substances indicated by the high concentration of COD, BOD, TDS, and phenol.

#### **EXPERIMENTAL**

#### Material and Methods

The pharmaceutical wastewater used in this study was obtained from PT X Indonesia located in Tangerang. The effluent has pH around 5 – 6 with the COD value around 1000 ppm and the initial phenol concentration was 4.75 ppm. The ozone was generated from the air by the ozone generator (Hanaco THS-278 type) with the dosage of 0.1 g/h. This dosage was obtained by Iodometric titration method. Granular activated carbon was purchased and it was applied without any further treatment. Commercial powder of TiO<sub>2</sub> P25 (79% anatase, 21% rutile) was supplied from Evonik Industries. Pumice is supplied from Bima City, Nusa Tenggara Barat, Indonesia. Tetra Ethyl Ortho Silicate (TEOS) or Si(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub> 98% and all chemicals used in this study were purchased from Sigma-Aldrich.

TiO<sub>2</sub> immobilized on pumice was performed using impregnation method. Pumice (PS) with the size of 4-6 mm was cleaned ultrasonically. Dip coating of PS in the sol of TiO<sub>2</sub> (5 % w/w of TiO<sub>2</sub> on PS) was performed with TEOS as an adhesive and Si source. Finally, the composite PS-TiO<sub>2</sub> was evaporated in the furnace at 350 °C for 1 h. The PS-TiO<sub>2</sub> was characterization by FESEM/EDX (FEI inspect F50) and then it underwent photocatalyst test for degrading pharmaceutical wastewater.

#### Batch-recirculation reactor system and wastewater treatment procedure.

The configuration of two batch circulation reactors with ozonation and photocatalytic processes can be seen in Fig. 1. The waste from the wastewater tank that made of glass with the volume of 30 L (length 30 cm, width 30 cm, and height 34 cm) was firstly pumped through the pipe (ID = 2.1 cm) and it was injected with O<sub>3</sub>/O<sub>2</sub> from the ozone generator with the O<sub>3</sub> producing rate 0.1 g/h. Subsequently, the waste went through the two carbon columns and finally, it experienced photodegradation in the 30 L of the photoreactor that equipped with the mercury lamp (HPL-N 250 W/542 E40 HGISL) as a photon source (17% of UV and 83% of visible light). For the ozonation, the schematic process was similar to the Figure 1, however, the flow after through GAC was directly recirculation to wastewater tank. For the photocatalysis, the wastewater was allowed to flow directly to photoreactor and back to the wastewater tank. The circulation flow rate of the wastewater was setup and variated (10; 12; 15) L/min, whereas the amount of the PS-TiO<sub>2</sub> also variated (200; 250; 300) g. At a time interval, the COD and phenol concentration were measured according to SNI and ASTM standards.

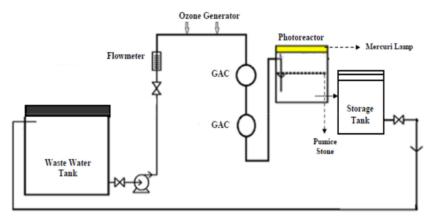


FIGURE 1. The experimental setup for ozone process and photocatalysis

#### RESULT AND DISCUSSION

#### Photocatalyst characterization

The FESEM image of PS-TiO $_2$  indicated that PS has pores and the surface has covered by TiO $_2$  as shown in Fig. 2.

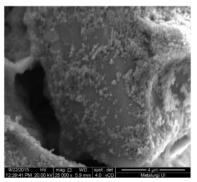
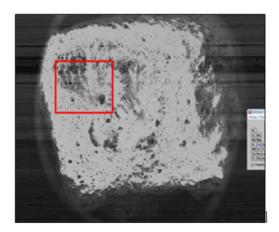


FIGURE 2. FESEM image of PS-TiO2 (magnification of 25000 X)

The EDX results of PS-TiO<sub>2</sub> (Table 1 and Fig. 3) indicated that besides O and Ti, the components like Na, Al, Si, K, and Ca that contained in PS were detected and it gave evident the successful impregnation of TiO<sub>2</sub> on PS. Similar result also reported by previous researchers [10,12,13]. Table 1 presents the elemental composition of PS-TiO<sub>2</sub>.

Table 1. Elemental composition of PS-TiO2

Elements	%Weight
О	63.1
Na	2.7
Al	3.69
Si	11.91
K	0.63
Ca	0.33
Ti	17.2



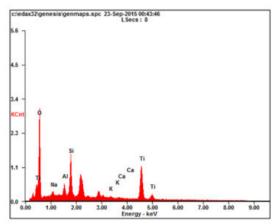


FIGURE 3: EDX analysis of PS-TiO2

#### Pollutants degradation by ozone process

Figure 4 shows the reduction of COD from wastewater by  $O_3/GAC$  as a function of time with 300 g of PS-TiO<sub>2</sub> at the different circulation flow rate. The reduction of COD is caused by the oxidation of the pollutant with: a). O<sub>3</sub> (ozonolysis), b). with hydroxyl radical (\*OH) results from the reaction of O<sub>3</sub> with water, and c). the reaction of O<sub>3</sub> in the surface of GAC. The function of GAC is not only as the adsorbent that removes organic carbon, but also accelerate the O<sub>3</sub> transformation into OH [5]. As a result, ozonation process is very effective in the degradation of pollutant in wastewater. The high performance of ozonation is caused by the high oxidative power of hydroxyl radical and ozone with the redox potential of 2.8 V and 2.07 V respectively [1,4]. There are two reactions through which O<sub>3</sub> decomposes the pollutant. Firstly, direct attack of pollutants by molecular ozone (ozonolysis) that occurs at pH bellow 7 (acidic or neutral condition). This condition is the selective reaction that results in the end product that can't be oxidized further by O<sub>3</sub>. Secondly, nonselective decomposition of pollutants by OH radicals at high pH value [4]. It is clear that the circulation flow rate of 10 L/min of the wastewater provides the highest COD removal since at this condition the contact time and the mixing of O3 and OH with the pollutant in waste water are sufficient and better compared to others. As a result, pollutant could be degraded effectively. This result is in accordance with the previously reported by Linda Zou et al. (2008) when they studied color removal from reused water with ozone and photocatalytic process [14]. To fulfill the COD standard quality requirement (bellow 300 ppm), this process needs around 12.6 h. For phenol degradation, it diminished from 4.75 ppm to become 0.1 ppm in 6 h ozonation process. On the other hand, at higher circulation flow rate, the contact time of ozone and hydroxyl radical to the pollutant is decreased, which resulted in the slower COD removal. The reduction of COD and phenol concentration due to the oxidation of pollutant in the pharmaceutical wastewater to become degradation product by O<sub>3</sub>, \*OH, and adsorbed by GAC.

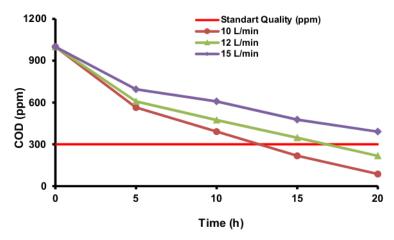


FIGURE 4. The COD reduction as a function of time with ozonation process for various circulation flow rate

#### Pollutants degradation by photocatalysis

Figure 5 depicts COD removal of pharmaceutical wastewater as a function of time irradiation at 10 L/min circulation flow rate with different amount of photocatalyst. It is clear that the COD rapidly reduced by the photocatalytic process with 300 g of Ps-TiO<sub>2</sub> compared to others. In this process, the hydroxyl radical is considered as the main substance that responsible for degradation of organic matters [11, 14]. In photocatalysis, when TiO<sub>2</sub> absorbs photon greater than its bandgap energy, there is excitation electrons from valence band (vb) to conduction band (cb) and therefore, resulting electrons in the conduction band and holes in the valence band according to Eq. 1. The holes react with water to produce OH (Eq. 2), and this OH and the holes itself oxidizes the pollutants to become CO<sub>2</sub> and H<sub>2</sub>O [4,15]. Hole is the highly oxidizing agent (+2.53 eV versus standard hydrogen electrode, SHE) that effectively degrade the pollutant [16]. Meanwhile, the electrons can act as the reducing agent to remove heavy metals such as mercury, arsenic, and chromium [17] or reacts with the adsorbed oxygen on the surface of PS-TiO<sub>2</sub> to form hydroxyl radical according to Eq. 3 through various pathways, [4,11]. The reactions in the photocatalysis are as follow.

$$\overline{TiO_2} + hv \rightarrow TiO_2 + e_{cb}^- + h_{vb}^+ \tag{1}$$

$$h_{vb}^{+} + H_{2}O \rightarrow {}^{\bullet}OH + H^{+}$$

$$h_{vb}^{+} + OH^{-} \rightarrow {}^{\bullet}OH \tag{2}$$

$$e_{cb}^{-} + O_2 \to O_2^{\bullet -} 2O_2^{\bullet -} + 2H_2O \to 2^{\bullet}OH + 2OH^{-} + O_2$$
(3)

The pollutants (P) that adsorbed in the surface of PS-TiO<sub>2</sub> plays an important role in the photocatalytic oxidation via successive attacks by OH or hole as follows [14].

$$P + {}^{\bullet}OH \rightarrow P^{\bullet}, + H_2O$$
 (4)

$$P + h_{vb}^+ \rightarrow P^{\bullet +} \rightarrow degradation \ product$$
 (5)

From Fig. 5 it can be stated that the amount of PS-TiO<sub>2</sub> significantly affects the COD removal. The more the amount of PS-TiO<sub>2</sub>, the higher the COD removal and 300 g of the photocatalyst in this process result in the highest COD removal. This condition is considered as the optimum process. It meant that more \*OH and hole are responsible in degrading pollutant. This photocatalysis needs 12 h to fulfill bellow the COD standard quality requirement (290 ppm) that allowed to discharge in the environment. In this condition, the percentage COD removal was 71 %. To reduce the time of the process, the combination of ozonation and photocatalysis was performed.

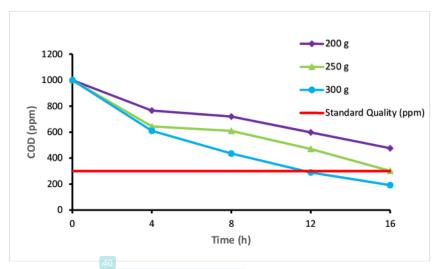


FIGURE 5. The COD removal as a function of time with photocatalytic process for various PS loading

#### Pollutants degradation by combination of ozone process and photocatalysis

The combination of ozonation followed by photocatalysis processes with batch circulation reactors was performed with the equipment that can be seen in Fig. 2 at the flow rate of 10 L/min and 300 g of the photocatalyst. The PS-TiO<sub>2</sub> covered the surface of the liquid waste in the photoreactor. From Fig. 6, it is clear that the COD removal was rapidly degraded by the combination of the two methods compared to the degradation by the photocatalysis or ozonation alone. The result indicated that after 4 h circulation, the initial COD and phenol concentration of around 1000 and 4.75 ppm declined to 290 and 0 ppm, respectively. On the other hand, the photocatalysis or ozonation alone only reduces the COD to 609 and 651 ppm, respectively. This result is in a good agreement with the study that previously reported [11,14]. In ozone process, O<sub>3</sub> and hydroxyl radicals are responsible for pollutant decomposition. However, electrons generation by photocatalytic also could react with ozone to product \*OH via several pathways [14] as follows.

$$O_3 + e_{cb} \longrightarrow O_3 \longrightarrow OH$$
 (6)

The availability of O<sub>2</sub> introduced via ozone generator also can react with the electron to become •OH as final products (Eq. 3). As a consequence, a larger number of •OH (powerful oxidant species) can be produced by the combination of those two processes. In another word, the rapid degradation using this combination process due to not only the role of O<sub>3</sub>, •OH, GAC in ozonation and the influence of •OH and hole that resulted from photocatalysis process, but also the increasing the number of radical hydroxyls. As a result, the combination process results in more powerful oxidant that responsible for degrading pollutant. The use of combined ozonation and photocatalysis is an attractive route because of the enhancement of the hydroxyl radical generation, a powerful oxidant agent that can oxidize completely the organic matter present in the aqueous system. Ozone process is reported to be efficient for highly contaminated wastewater such as pharmaceutical waste, meanwhile, photocatalysis can completely decompose the intermediate product that resulted from ozone process. Moreover, photocatalysis also found to be efficient to degrade the dilute solution.

Compared to the ozone process, the photocatalytic process indicated slightly higher COD removal. It has been reported that, most of the organic matters could not completely decompose by ozone alone [14]. Moreover, the pH of this treatment was around 5-6, and therefore, pollutant was decomposed mainly by direct attack of  $O_3$  and it is a selective reaction that resulted in intermediate product. Since this intermediate product cannot be oxidized further, the COD value was slightly higher [4].

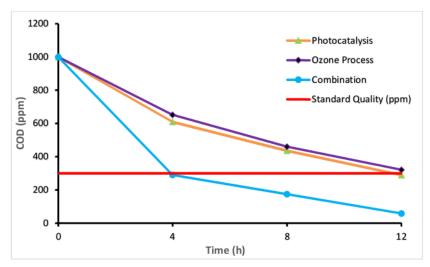


FIGURE 6. The COD removal as a function of time with variation process

#### CONCLUSION

The combination of ozone process and photocatalysis in a batch reactor with circulation shows as an effective method for pharmaceutical wastewater treatment with relatively high contaminated wastewater indicated by the COD value of 1000 ppm. This process can treat 20 L of pharmaceutical wastewater with the initial COD of 1000 ppm and phenol concentration of 4.75 ppm decrease to 290 ppm and 0 ppm, respectively for 4 h with the circulation flow rate of 10 L/min and 300 g PS-TiO<sub>2</sub>. This condition meets the standard quality of effluent and it is considered as an optimum process. Ozone and photocatalytic processes could be an effective and alternative treatment for pharmaceutical wastewater with high concentration of pollutants.

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