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## Kinetic Study of Paracetamol Degradation with Advanced Oxidation Process (AOP) Combination of Ozone, Hydrogen Peroxide and Ultraviolet (O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>/UV)

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### ABSTRACT

Paracetamol is an analgesic and antipyretic drug commonly used by the public, with consumption reaching thousands of tons per year. Paracetamol, also known as acetaminophen, consists of a benzene ring core substituted by a hydroxyl group and a nitrogen atom. Paracetamol is not easily adsorbed or biologically degraded, which raises significant concerns about its impact on humans and the environment. One of the commonly used conventional treatments for paracetamol involves chlorine, which produces hazardous by-products such as 1,4-benzoquinone and N-acetyl-p-benzoquinone imine. Therefore, a better and safer method is needed for the treatment of paracetamol. Advanced Oxidation Processes (AOPs) are proven methods for treating difficult-to-degrade organic compounds and converting them into simpler compounds. AOPs utilize free radicals to oxidize pollutant compounds, transforming them into more manageable forms. The performance of AOPs can be enhanced by combining oxidants such as ozone, hydrogen peroxide, and ultraviolet light. Hence, in this research, the AOP method is employed to treat paracetamol, and the study aims to analyze the kinetics, efficiency, and by-products of this AOP method

## INTRODUCTION

Drugs from pharmaceutical products have been used intensively by humans for decades and their presence is often detected in wastewater, freshwater and coastal areas (Gaw et al., 2014; Aus Der Beek et al., 2016., 2016) . Paracetamol is a pharmaceutical product that is widely consumed, especially during the COVID pandemic. Paracetamol is an analgesic and antipyretic drug that is very commonly consumed, which can reach thousands of tons per year (Sebastine and Wakeman, 2003). Paracetamol is also known as acetaminophen, 4-acetamidophenol, N-(4 hydroxyphenol) acetamide (C<sub>8</sub>H<sub>9</sub>NO<sub>2</sub>), and consists of a benzene ring core substituted by one hydroxyl group and a nitrogen atom from the amide group in a para (1,4) pattern. These pharmaceutical products are often referred to as "emerging pollutants" because they produce new problems due to the lack of available information about their impact on the environment or interference with biological processes (Fent et al., 2006). Many of the active substances from these pharmaceutical products are persistent because they are transparent to conventional wastewater treatment and therefore disperse into the environment (Carballa et al., 2004; Tauxe-Wuersch et al., 2005). Therefore, there is great concern about the impact of pharmaceutical compounds on public health and the environment, not only because of their acute toxicity, but also their genotoxicity as well as the development of pathogen resistance and endocrine disruption (Halling-Sørensen et al., 1998; Graham et al., 2011 )

Paracetamol is not only detected in hospital waste which is considered a source of paracetamol pollution, but also in wastewater treatment, rivers and sludge with concentrations ranging from 6-65 µg/L (Bound and Voulvoulis, 2006; Gómez et al., 2006; Kinney et al., 2006; Thomas et al., 2007; Al Rifai et al., 2007). Although paracetamol is biodegradable (Sang et al., 2007), its degradation rate is slow and does not allow complete elimination with conventional wastewater treatment plants in general. On the other hand, processing with chlorine which is usually used as a disinfectant is known to convert paracetamol into toxic intermediates such as 1,4

benzoquinone and N-acetic-pbenzoquinone imine (Bedner and Maccreehan, 2006).

Paracetamol does have a high level of degradation, but in reality traces of it are still found in liquid waste up to 200 µg/L (Togola and Budzinski, 2008), 0.101-20.86 µg/L in wastewater in Kuwait (Alajmi, 2014), in United Kingdom detected more than µg/L in the river Tyne (Roberts, 2006), in wells supplying drinking water detected 0.211 µg/L (Rabiet, 2006). In Indonesia itself, paracetamol has been detected in quite high concentrations in the Angke estuary (Koagow, 2021). the study on acetaminophen (ACT), a compound similar to paracetamol, highlights the generation of various by-products and their biotoxicity, emphasizing the importance of understanding the degradation pathways and by-products in the treatment process (Muhammad, 2022). The combined US/H<sub>2</sub>O<sub>2</sub> technology, while not directly applied to the O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>/UV combination, illustrates the color changes and by-product formation during the oxidation of paracetamol, suggesting the complexity of reactions involved in AOPs (Villota, 2022). The Fe(III)/S(IV)/O<sub>2</sub> system under UVA irradiation, although not directly related to the O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>/UV combination, showcases the potential of using irradiation to enhance degradation processes at near-neutral pH, which could be relevant for optimizing paracetamol degradation conditions (YaNan, 2022). The catalytic properties of iron ions in the oxidative destruction of paracetamol with hydrogen peroxide further support the effectiveness of combining oxidative agents with catalysts to achieve high degradation efficiency (Adrian, 2022).

Several previous studies have studied the degradation of paracetamol in water using advanced oxidation processes such as radiolysis, sonolysis, catalytic wet air oxidation (CWAO) in activated carbon, ozonation, and photolysis (Dalmázio et al., 2008; Yang et al., 2008; Andreozzi et al., 2003; Quesada Peñate et al., 2009b, 2012; Neamtu et al., 2013; Aguinaco et al., 2014; Torun et al., 2014). The advanced oxidation process is a processing method that uses oxidants to reduce them directly or can form free radicals which can later be used as oxidants.

Ozone, hydrogen peroxide and ultraviolet are the most commonly used oxidants for this method. This method can also be maximized with a combination of oxidants so that it can produce oxidants in the form of free radicals that are greater than those used alone. Therefore, paracetamol is processed using a combination of ozone, hydrogen and ultraviolet ( $O_3/H_2O_2/UV$ ) oxidants to increase its degradation and see how the oxidation results from the processing that has been proposed.

## METHODS

### Preparation

Experiments on the advanced oxidation process will be carried out on a laboratory scale. The location of the research will be carried out at the Water Laboratory, Environmental Engineering, ITB. The oxidant used in this research is ozone produced by an ozone generator, UV produced by a 20Watt UV lamp and 30% hydrogen peroxide. The waste used is synthetic waste which is made by dissolving raw paracetamol powder in NaOH solution.

Research on further oxidation of paracetamol was carried out using a laboratory scale semi-batch technique using a column reactor (Figure 1). The waste to be processed has a volume of 1 liter with a paracetamol content of 200mg/L.

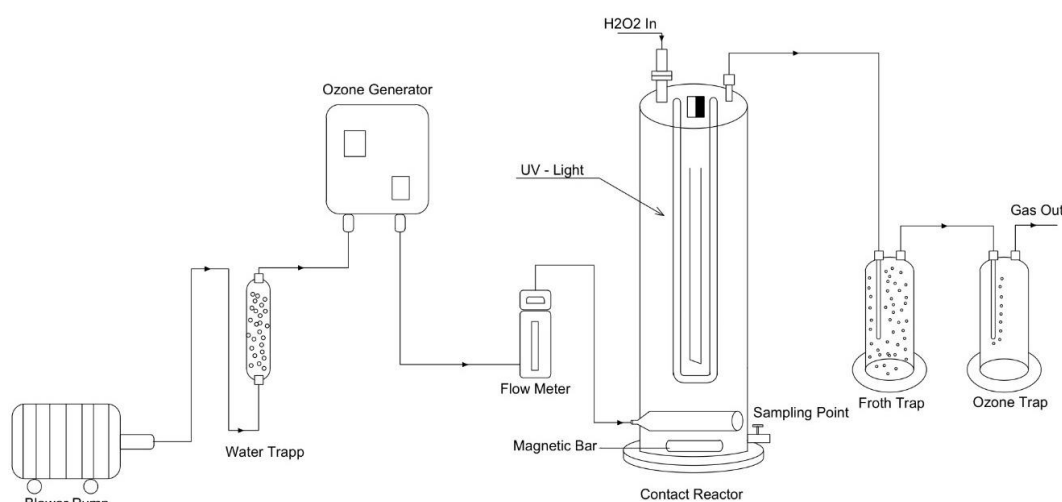


Figure 1. Reactor Scheme

### Paracetamol Oxidation

Experiments were carried out by varying the pH, ozone flowrate and hydrogen peroxide dosage. The experimental set is a modification of research conducted by Rohman 2016. In this research, only two oxidants were used, namely hydrogen peroxide and ozone and a contaminant in the form of 4-chlorophenol. During the further oxidation process, pH, temperature and paracetamol levels were checked and additionally checked for color, conductivity and turbidity at the beginning and end of the experiment.

### pH Regulation

In this experiment, pH was adjusted by adding  $H_2SO_4$  and NaOH, where the pH used was 3, 7, and 10 which describes the acidic, basic and neutral conditions of wastewater. pH adjustment is carried

out because it can influence the formation of free radicals by the proposed oxidant.

### Flowrate Ozone

The ozone flowrate variations used in this experiment were 0.5 lpm, 1 lpm, and 1.5 lpm. This flowrate variation refers to previous research conducted by Rifai (2015). From the selected ozone flowrate variations, the ozone-feeding rate will be tested to see how much ozone is dissolved per minute. This test is carried out using iodometric and spectrophotometric methods.

### Dosage of Hydrogen Peroxide

In this experiment, the dose of hydrogen peroxide was adjusted, where the doses used were 500ppm, 1000ppm and 1500ppm. The choice of this variation refers to previous research which used a hydrogen peroxide dose of 600ppm to 2000ppm González (2016).

### Residual Hydrogen Peroxide

Testing of the remaining hydrogen peroxide after the oxidation process was carried out to see how much hydrogen peroxide was decomposed using the H<sub>2</sub>O<sub>2</sub>/UV method. This method is carried out by titration method with potassium permanganate (KMnO<sub>4</sub>).

### Combination

The combination is carried out by combining both oxidation methods, namely ozone and hydrogen peroxide with UV (O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>/UV). The combination is carried out by selecting the optimum conditions for each oxidant to be tested at each proposed pH.

### Analysis of Paracetamol Levels

Analysis of paracetamol levels using the HPLC-DAD method. This analysis is carried out on waste samples. In this method, the mobile phase is used: methanol: aquabidest (70:30) (v/v), stationary phase: C18 column (4.6 mmid x 250mm), flow rate: 1.0 ml/minute, injection volume: 20 µL, detector: UV at wavelength 256 nm.

### Analysis of Oxidation Results

Analysis of oxidation results was carried out using the GCMS method. This method was carried out to see changes in paracetamol after going through a further oxidation process using the proposed method.

### Kinetic Analysis

Kinetic analysis is carried out by testing samples at each predetermined time range. Where in this research samples were taken every 15 minutes

with a processing time of 2 hours. This analysis is to see how the reaction rate constant is obtained from each variation that will be carried out in the experiment.

### Statistic Analysis

The data obtained will be explained descriptively using Analysis of Variance (ANOVA) to compare the mean/median of different treatments. Statistically, processing with a P value of less than 5% is considered a significant factor that may influence the degradation rate of paracetamol.

## RESULTS AND DISCUSSION

### Ozone

O<sub>3</sub> is a toxic and unstable gas that is quickly converted to O<sub>2</sub>. For single ozonation and other ozonation treatments to remove PCT from water and wastewater, O<sub>3</sub> must be produced continuously on-site by a generator after injection of air or pure O<sub>2</sub> on one side of the device. Ozone can react with substances directly as O<sub>3</sub> or indirectly as free radicals. Figure 2 shows that there are differences in the percentage of PCT removal by ozone at different pH levels. The best PCT removal percentage occurred at pH 10 where PCT removal could reach 100% at a retention time of 90 minutes. The difference in pH in the ozonation process affects the reaction of ozone to pollutant substances, where at pH 10 ozone turns into free radicals in reducing pollutant substances.

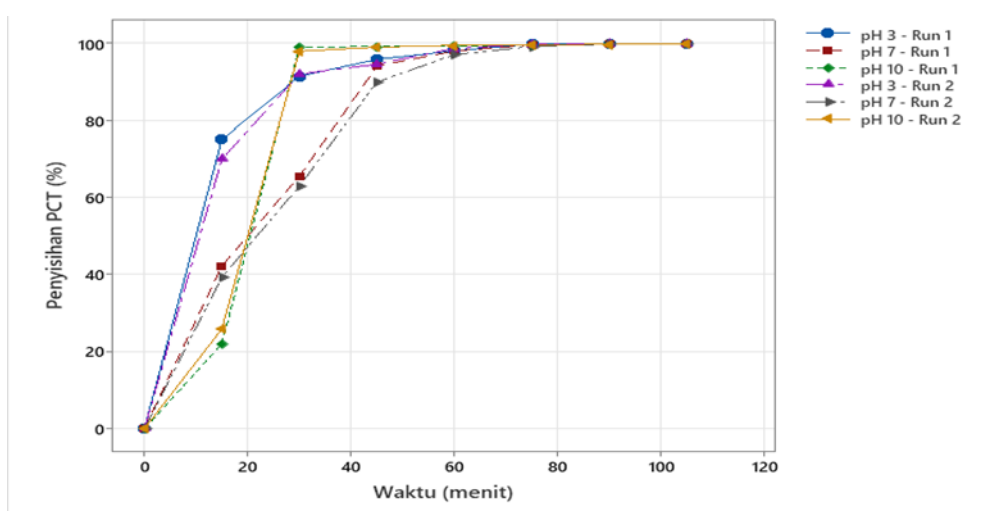


Figure 2. PCT Removal Percentage at Ozone-feeding Rate 58.39 PPM/Minute

### H2O2/UV

The H2O2 Photolysis process is an advanced oxidation process that utilizes ultraviolet as an energy source in decomposing H2O2 into hydroxyl radicals ( $\text{OH}\cdot$ ). The hydroxyl radicals formed from this process are used as oxidizing agents to reduce pollutants.



Figure 3 shows the differences in the percentage of PCT removal through different pH conditions, where the optimal pH for removing PCT in this process is at pH 10 which can reach 79% at a retention time of 120 minutes.

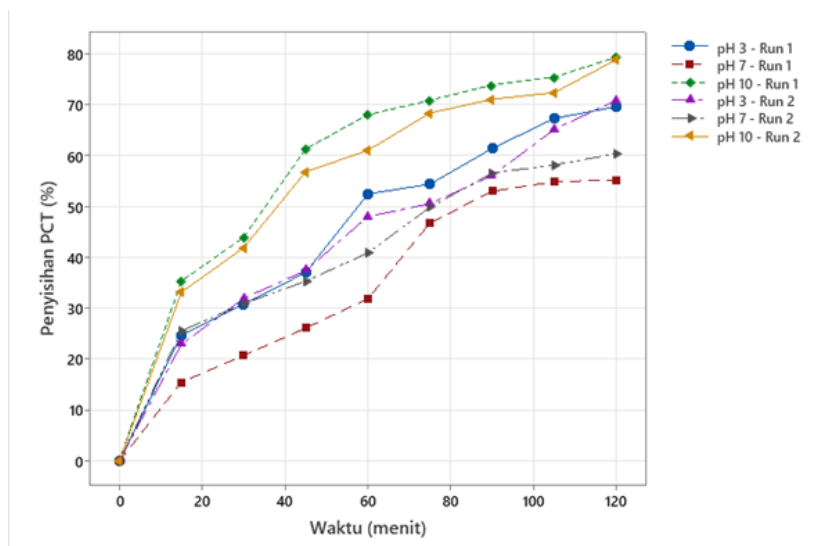
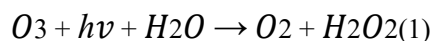


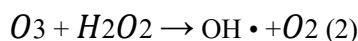
Figure 3. Percentage of PCT Removal at a H2O2 Dose of 1500 PPM

### O3/UV

Based on the results of ozonation processing without a catalyst, additional experiments were carried out on O3 with the addition of a catalyst in the form of ultraviolet with a wavelength of 254nm. Compared with the ozonation process alone, the O3/UV combination showed a percentage increase in PCT removal. The O3/UV combination accelerates PCT removal efficiency, where the removal percentage can reach 100% at a retention time of 60 minutes. The chemical reaction can be summarized as follows



The peroxide then reacts with ozone to form hydroxyl radicals



### O3/H2O2

The O3/H2O2 method has been widely used and is efficient in wastewater treatment, this method conjugated H2O2 groups can accelerate ozonolysis to produce more hydroxyl radicals ( $\text{OH}\cdot$ ). In this method, it can be seen that removal occurs more quickly compared to a single ozonation process, where the PCT removal efficiency can reach 100% at a retention time of 60 minutes.

### O3/H2O2/UV

The O3/H2O2/UV method is an oxidation process that is quite effective compared to other processes such as O3, H2O2/UV. In this process O3 is decomposed into hydroxyl radicals with the help of UV light and the addition of hydrogen peroxide causes an even better increase in reaction speed. This method showed a better increase in PCT removal than the O3, H2O2, O3/UV and O2/H2O2 methods. Where the PCT removal percentage can reach 100% at a retention time of 45 minutes.

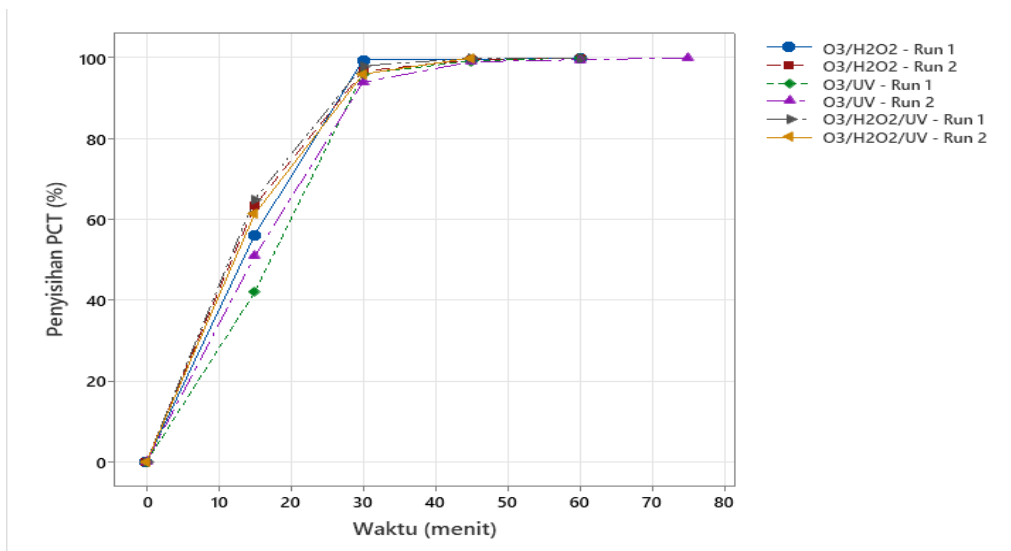


Figure 4. Percentage of PCT Removal in O3/H2O2, O3/UV and O3/H2O2/UV

Paracetamol is an organic compound, where the rate of degradation of organic compounds according to Ugurlu and Karaoglu, 2009 can be described through pseudo first order reaction kinetics with the following equation:

$$\ln[C_t]/[C_0] = -kt$$

The value of the reaction rate constant (k) is obtained by plotting a graph of the relationship  $\ln[C_t]/[C_0]$  as the y-axis and time as the x-axis. From this graph, a slope will be obtained which represents the value of the reaction rate constant (k). In this case,  $C_0$  is the initial PCT concentration (ppm) and

$C_t$  is the PCT concentration (ppm) at each time. The calculation results of the PCT degradation rate constant in Table 1 show that the dominant reaction rate constant is higher at 60 minutes. This shows that the PCT removal process is taking place more quickly, where this phenomenon can occur because the PCT concentration decreases over time but the oxidant concentration remains constant. From the results of ozone and H2O2/UV experiments, optimum conditions were obtained at an ozone-feeding rate of 58.39ppm/minute at pH 10 and an H2O2 dose of 1500ppm at pH 10.

Table 1. Calculation of PCT Degradation Rate Constants

Process		k' (menit <sup>-1</sup> )		
		60 minute	120 minute	
33,22 ppm/minute	Run 1	pH 3	0,0311	0,0359
		pH 7	0,0355	0,0467
		pH 10	0,0035	0,02
	Run 2	pH 3	0,0212	0,0212
		pH 7	0,0313	0,0313
		pH 10	0,0034	0,0034
Ozon	Run 1	pH 3	0,0563	0,0459
		pH 7	0,0362	0,0248
		pH 10	0,0394	0,0168
	Run 2	pH 3	0,0450	0,0475
		pH 7	0,0297	0,0272
		pH 10	0,0372	0,0021
41,49 ppm/minute	Run 1	pH 3	0,0649	0,0106
		pH 7	0,0677	0,0125

<b>H<sub>2</sub>O<sub>2</sub>/UV</b>	58,39 ppm/minute	Run 2	pH 10	0,0986	0,0106
			pH 3	0,0697	0,0107
			pH 7	0,0593	0,0138
			pH 10	0,1009 *	0,0054
	500 ppm	Run 1	pH 3	0,006	0,0076
			pH 7	0,0056	0,0063
			pH 10	0,0108	0,0085
			pH 3	0,006	0,0076
	1000 ppm	Run 2	pH 7	0,0056	0,0063
			pH 10	0,0108	0,0085
			pH 3	0,0081	0,0088
			pH 7	0,0056	0,0054
1500 ppm	Run 1	pH 10	0,0139	0,0094	
		pH 3	0,0094	0,0091	
		pH 7	0,006	0,0057	
		pH 10	0,0134	0,0089	
1500 ppm	Run 2	pH 3	0,0111	0,0098	
		pH 7	0,006	0,0072	
		pH 10	0,0187*	0,0123	
		pH 3	0,0101	0,0094	
1500 ppm	Run 2	pH 7	0,008	0,0074	
		pH 10	0,0155	0,0118	

From the optimum conditions obtained from single ozonation and H<sub>2</sub>O<sub>2</sub>/UV experiments, further tests were carried out by combining the optimum conditions to see the optimization that occurred when processing PCT. The combinations chosen in the follow-up test were O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>, O<sub>3</sub>/UV, and O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>/UV. From Table 2, it can be seen that the

combination treatment of optimum conditions shows an increase in the reaction rate constant (k). The best optimization of this combination is found in O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>/UV, where the reaction rate constant increases better than the ozone and H<sub>2</sub>O<sub>2</sub>/UV methods.

Table 2. Calculation of PCT Degradation Rate Constants

Process			k' (minute <sup>-1</sup> )			
			30 minute			
45 minute	60minute	120 minute				
<b>O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub></b>	Run 1		-	0,1415*	0,1014	0,0374
	Run 2		-	0,124	0,0748	0,021
<b>O<sub>3</sub>/UV</b>	Run 1	pH 10	-	0,1161*	0,0721	0,0214
	Run 2		-	0,1066	0,0982	0,0175
<b>O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>/UV</b>	Run 1		0,1308	0,1613	0,0404	0,0201
	Run 2		0,1703*	0,0151	0,0064	0,0103

#### Analysis of the Final Characteristics of Waste

Wastewater characteristics analysis was carried out in each process carried out in this research, where significant changes in the final characteristics of the

waste were obtained in the O<sub>3</sub>/UV process. The results of the analysis of the final characteristics of the waste can be seen in Table 3.

Table 3. Analysis of Final Characteristics of Waste Water

No	Parameter	Before	After	Unit
1	Color	14,2	1013,65	PtCo
2	pH	10	2,82	-
3	Turbidity	12,3	168,42	NTU
4	Conductivity	3,4	134	mS/m
5	Temperature	24,2	27,4	°C
6	Paracetamol	200	0	ppm

The results of analysis of waste water characteristics show significant changes, especially in the parameters of color, pH and turbidity. Increase in color parameters from 14.2 to 1013.65 PtCo, color change to yellowish in wastewater at the end of processing. Significant changes also occurred in the turbidity parameter, which before processing was 3.4 NTU to 168 NTU. This change in turbidity parameters occurs due to the formation of foam during the processing process so that the residue from the foam increases the turbidity in the final characteristics of the wastewater. The pH value also changed significantly where the pH condition was originally 10 to 2.82 at the end of processing, this could occur because of the possibility of the formation of acid products from the paracetamol processing process.

### CONCLUSION

The results of analysis of waste water characteristics show significant changes, especially in the parameters of color, pH and turbidity. Increase in color parameters from 14.2 to 1013.65 PtCo, color change to yellowish in wastewater at the end of processing. Significant changes also occurred in the turbidity parameter, which before processing was 3.4 NTU to 168 NTU. This change in turbidity parameters occurs due to the formation of foam during the processing process so that the residue from the foam increases the turbidity in the final characteristics of the wastewater. The pH value also changed significantly where the pH condition was originally 10 to 2.82 at the end of processing, this could occur because of the possibility of the formation of acid products from the paracetamol processing process.

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