Study of Application Advanced Oxidation Process (AOP) using Nano-Micro Bubble Ozone for Batik Industry Waste Water Treatment

Yudhistira Yossa Adirajasa[a], Ahmad Faruq Abdurrahman[a], Eko Yulianto[e], Sumariyah Sumariyah[b,e], Gunawan Gunawan[c], Sera Budi[d], and Muhammad Nur[b,,e*]
[a]Magisterof Physics, Physics Department, Diponegoro University, Tembalang Campus, Semarang, Indonesia, 50275
[b]Physics Department, Diponegoro University, Tembalang Campus, Semarang, Indonesia, 50275
[c]Department of Chemistry, Diponegoro University, Tembalang Campus, Semarang, Indonesia, 50275
[d]Doctoral Study Program Medical and Health Sciences, Faculty of Medicine, Diponegoro University
[e]Centerfor Plasma Research, Diponegoro University, Integrated laboratory, Tembalang Campus, Semarang Indonesia, 50275

Abstract:- Batik wastewater treatment using nano-micro bubble ozone has been carried out in this study. The batik wastewater used is rinsing wastewaster. The flow rate used in this study uses the best setting based on research data that has been done, which is 0.4 L/minute. Batik wastewater treatment was carried out for 300 minutes with sampling every 30 minutes. Waste samples were tested using UV-VIS to determine the absorbance value which indicates a decrease in waste parameters and the concentration of batik wastewater. The results showed that at 180 minutes, the waste had shown better color degradation which became clear and clean, then the treatment was continued for up to 300 minutes to obtain better parameter degradation. The removal efficiency is obtained by 74.43% and concentration of batik wastewater before treatment was 0.36 ppm. The EC, TDS, COD, and BOD parameters decreased significantly in proportion to the length of treatment, while the temperature increased with the length of treatment due to the pump engine getting hotter.

Keywords:- *advanced oxidation process, ozone, nano-micro bubble, batik wastewater.*

I. INTRODUCTION

Batik is a unique pattern on cloth that represents a very valuable heritage and icon in Southeast Asian countries, especially Indonesia. In 2009, Indonesian batik was recognized as a Masterpiece of Oral and Intangible Heritage of Humanity by UNESCO. Pekalongan is one of the largest cities in Indonesia as a batik producer. The increasing batik production in Pekalongan has a negative impact on the rivers in the batik industrial center area of Pekalongan. The impact is in the form of water pollution due to batik wastewater which is dumped directly into the river by batik craftsmen without being processed first so that the river water has an unpleasant odor and becomes colored [1,2,3]. For example, the flood in Pekalongan on February 2021 which went viral on social media because of the red flood water which is thought to be the cause of the red color due to the batik waste that was disposed of being stored in a certain place around the factory before being processed.

Batik wastewater is generally characterized by alkaline pH, striking color, high chemical oxygen demand (COD) and biological oxygen demand (BOD) [4]. Batik wastewater is toxic to the surrounding environment because in addition to containing dyes, batik wastewater also contains synthetic materials that are difficult to degrade. Several studies have reported that batik wastewater contains compounds such as wax oil, heavy metals, surfactants, suspended solids (SS), phenol, chromium, and organic and inorganic dyes [5, 6, 7, 8]. These dye compounds are quite stable and difficult to degrade, so that in high concentrations these compounds are harmful to the environment because they able to increase the levels of COD and BOD in water [5]. Synthetic dyes in batik wastewater able to cause skin problems such as skin irritation, dermatitis, allergies, and cancer [9,10]. The dark color on batik wastewater able to block sunlight from entering the water flow so that it can disrupt the ecosystem in the river [11]

Based on the Minister of Environment of the Republic of Indonesia No. 6 of 2021 which states that, "a waste must meet the specified quality standard value to be released into the environment" meaning that waste discharged into the environment must go through a certain process to reduce the level of toxicity of the waste so that it does not have a harmful or less harmful impact on the environment. The process of handling batik wastewater mostly uses filtration and adsorption processes either physically, chemically, or biologically such as coagulation, sedimentation, adsorption, and electrolysis [12]. In addition, there are other ways of processing wastewater, one of which is by increasing the advanced waste oxidation process or what is commonly called the Advanced Oxidation Process (AOP) [13]. AOP is a set of ways to improve the oxidation process in sewage where the production of hydroxyl radicals is a common denominator which one of the basic types of AOP can be through ozonation or ozone based [14]. Ozone itself is a strong oxidizing agent and has high color removal efficiency and is effective at high pH values [15,16]. In addition to the coagulation process, to produce ozone, dielectric barrier discharge (DBD) can also be used, namely with O2 gas, CO2, and ambient air as input gas using two electrodes that carry high electric currents [17, 18, 19, 20]. If only using the ozonation process, for example for dispersive dyes, it will be less effective, therefore a combination of the ozonation process is needed to achieve the desired purpose [21,22].

The use of ozone which is applied directly to methylene blue textile waste can reduce the content of wastewater parameters [23]. However, this still less effective, namely the relatively low mass transfer value [13, 24, 25]. To solve this problem, micro bubble technique can be used which is believed to be able to increase mass transfer so that the wastewater treatment process becomes more optimal. Microbubbles are small bubbles with a radius of less than 25 µm which are characterized by large specific surface area, long residence time, high mass transfer efficiency, and large release of hydroxyl free radicals in water [26, 27]. The use of microbubbles can improve mass transfer, mixing efficiency, energy efficiency, and reduce carbon footprint [28].

Microbubble ozone has the ability to reduce the color of textile wastewater [13, 29, 30], decompose organic pollutants [31], and remove particles or oil from the liquid phase [32]. Research conducted by Athikoh et al. [13] showed that microbubble ozone was able to reduce the indigo dye in textile wastewater to be clearer with an ozone capacity of 86.4 grams/hour for 540 minutes. Mass transfer, color reduction, mineralization and ozone accumulation for textile sewage treatment practices, microbubble systems hold promise for improving ozonation efficiency [30].

This study treats rinsing batik wastewater with reactive red dye 195 using nano-micro bubble ozone. Waste treatment time variations were carried out in this study to obtain the optimum time in degrading the rinsed batik wastewater to be clearer so that it is safe to be disposed of into the environment or reused.

II. RESEARCH METHODS

In Batik wastewater used as a sample is batik wastewater belonging to Batik Nurkis UMKM located in North Pekalongan. The 20 liter sample used was treated using nano-micro bubble ozone for 300 minutes with sampling every 30 minutes. The ozone generator used is a Dielectric Barrier Discharge (DBD) reactor, the flow rate used is 0.4 L/minute with an ozone concentration of 720 mg/L and a capacity of 17.28 grams/hour. The type of dye used is reactive dye/remazol red 195 ($C_{31}H_{19}ClN_7Na_5O_{19}S_6$) polan brand. The chemical structure of the reactive dye red 195 is as follows.

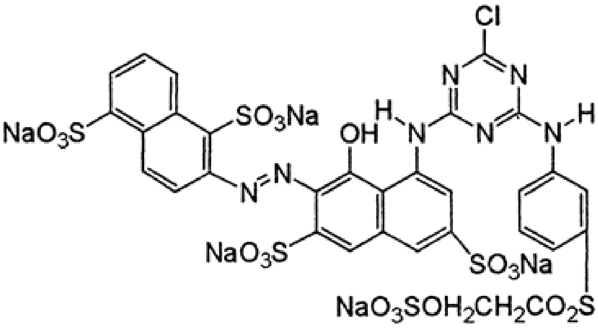


Fig. 1: Chemical structure of dye reactive red 195.

The control characteristics of the waste before testing are presented in Table I.

Parameters	Control
TDS	563 ppm
EC	1131 µs/cm
pH	7
Temperature	26 °C
COD	61.474 mg/l
BOD	19.057 mg/l

Table 1: Characteristic of batik wastewater with dye RR 195

The set-up of the equipment and configuration used in this study is depicted in Fig.2.

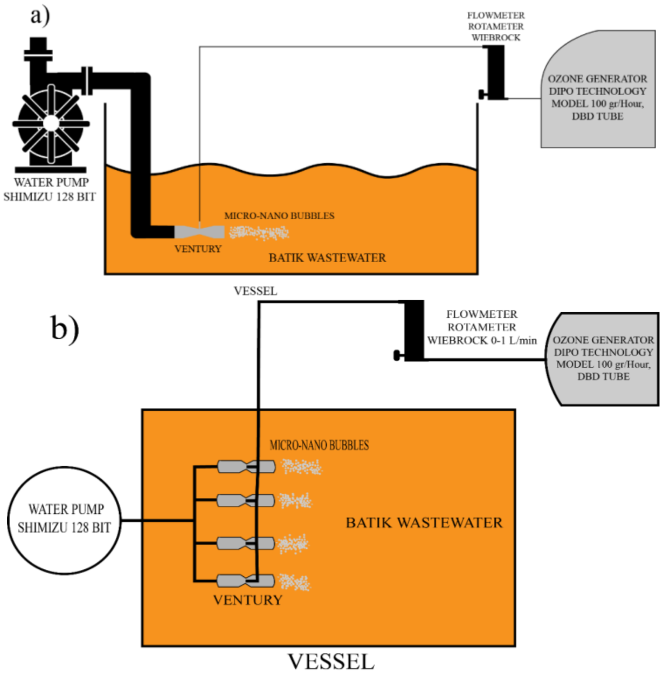


Fig. 2: Set-up Equipment (a) side view and (b) top view

The water pump is used to draw water and discharge water in the same place so that flow occurs. At the same time, the venturi pipe was fed with ozone at a flow rate of 0.4 L/min. This is because it uses the best settings from the previous experiment. In addition, the smaller the flow rate used, the smaller the diameter of the bubbles produced, meaning that the production of micro and nano bubbles is increasing [33, 34, 35]. Installation of venturi pipes in parallel is better when compared to installation in series. This is because the installation of venturi pipes in series is almost the same as single venturi pipes and does not depend on the number of venturi pipes installed in series [35].

Dissolve Ozone (DO) is the amount of ozone dissolved in a liquid. Measurement of ozone solubility using a tool*spectroquant colorimeter move O_3 Made Germany* 0,02 – 3,00 mg/O_3 with ozone mode setting 5. The reagents used are O_3-1 potassium iodide and O_3-2 boric acid. The medium for dissolving ozone uses 25 liters of distilled water. Sampling every 20 minutes for 1 hour is 10 ml then given 2 drops O_3-1 and 1 tablespoon O_3-2 Then the solution was allowed to react for 1 minute.

Removal Efficiency is how much waste has been cleared or removed by comparing the control waste minus the waste after treatment with the control waste. Efficiency calculations are obtained by looking at graphs in certain areas in UV-VIS and the results of efficiency calculations are usually in the form of percentages(%). The amount of cleaning efficiency can be calculated using the formula (1) [36]:

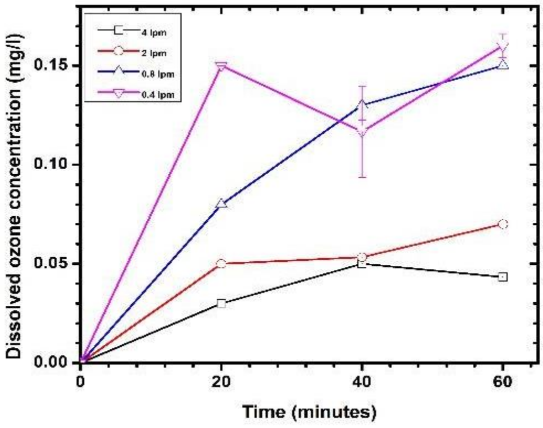
$$Removal\ Efficiency\ (\%) = \frac{C_0 - C}{C_0} \times 100 \qquad (1)$$

Where C_0 is control parameter (Abs) and C is parameter after treatment (Abs).

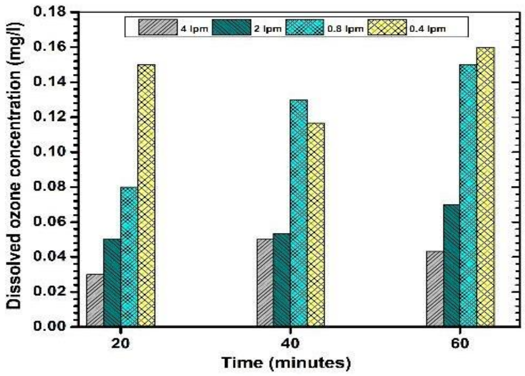
III. RESULT AND DISCUSION

A. Ozone Solubility

The ozone solubility value in this study was obtained by using micro and nano bubble ozone into distilled water for 60 minutes with an interval of 20 minutes. This research used 4 venturi pipes which are installed in parallel as nano-micro bubble generator.



(a)



(b)

Fig. 3: (a) The trend of the ozone solubility concentration as a function of time and (b) bar graph of ozone solubility concentration as a function of time

Distilled water of 25 Liters has been used in this study to determine the solubility of ozone. The ozone solubility obtained at flow rate of 4 L/min was 0.03 mg/L after 20 minutes; 0.05 mg/L after 40 minutes; 0.04 mg/L after 60 minutes. Flow rate 2 L/min obtained ozone solubility of 0.05 mg/L after 20 minutes; 0.05 mg/L after 40 minutes; 0.06 mg/L after 60 minutes. Flow rate 0.8 L/min obtained 0.08 mg/L ozone solubility after 20 minutes; 0.13 mg/L after 40 minutes; 0.15 mg/L after 60 minutes. Flow rate 0.4 L/min obtained ozone solubility 0.15 mg/L after 20 minutes; 0.09 mg/L after 40 minutes; 0.16 mg/L after 60 minutes.

The resulting ozone solubility increases with time and tends to be stable for a long time. The flow rate used in this study was 0.4 L/min. This is because the use of ozone solubility results is more optimal when compared to larger air flows. That is, the greater the flow rate used, the lower the solubility of ozone produced. The use of a smaller flow rate provides more effective results when compared to a larger flow rate [11].

The linear regretion has been done to analyze the relation between dissolved ozone concentration and time. The result has shown that time has a significant effect to the dissolved ozone concentration as the fig.4 shown below. This is evidenced by the trend of increasing ozone solubility over time.

This can happen because the ozone trapped in the microbubble bursts in the water and does not rise into the air. The high pressure on the microbubbles makes the ozone not easy to break and increase the solubility overtime.

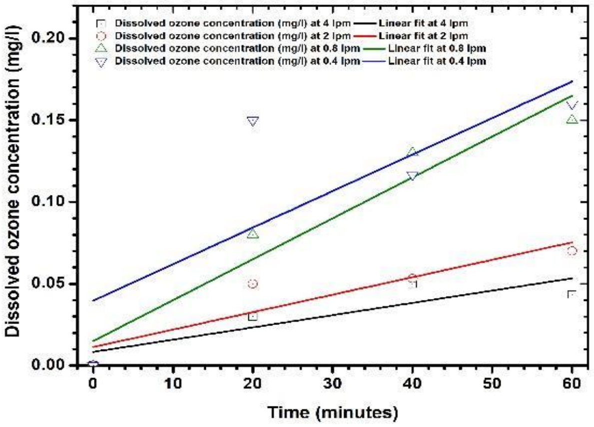


Fig. 4: Flow rate affect on Dissolved Ozone

B. *The Influence of Flowrate on Ozone Concentration and Capacity*

The characterization of the ozone generator was charged using high voltage source of 10 kV. In the process when ozone has been produce, high energy needed to dissociate the Oxygen to produce O atoms. Then, the O atoms hit the O_2 and these O atoms recombined in a three-body reaction to produce ozone [19].

$$e^- + O_2 \rightarrow 2O + e^- \tag{2}$$

$$O + O_2 + O_2 \rightarrow O_3 + O_2 \tag{3}$$

The effect of flowrate on ozone concentration and capacity is given by Fig.3. The maximum ozone concentration was obtained when gas flowrate was 0.4 L/minute at 0.72 ppm and the lowest value when the gas flowerate was 4 L/minute at 0.1 ppm. The increase in the gas flowrate results lower ozone concentration and on the contrary the decrease in the gas flow rate generate the higher ozone concentration.

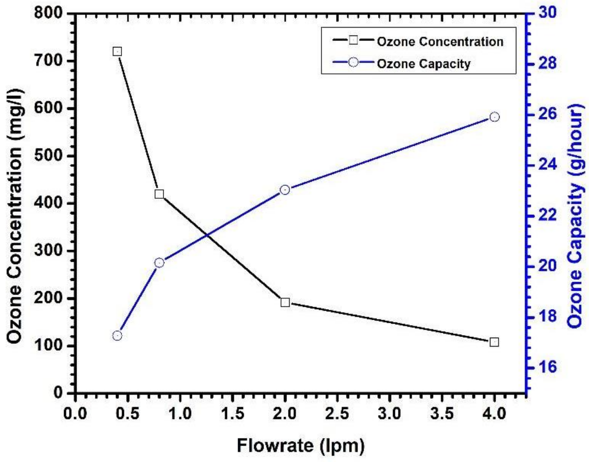


Fig. 5: The Effect Of Flowrate In Ozone Concentration And Capacity

Ozone capacity is defined as the number of produced ozone in a several time. Fig.5 shows that ozone capacity is directly propotional to gas flow rate. The maximum ozone capacity was 25.92 gram/hour when the input gas was 4.0 L/minute. The same thing was also obtained by Zahar*et al.* [37] That the ozone capacity is proportional to gas flow rate in constant voltage.

C. *Ozone Solubility Relationship with Ozone Capacity and Concentration*

Ozone capacity and concentration of course affect its solubility in water. As shown in fig.6, the graph of the relationship between the concentration of ozone solubility and its concentration and capacity. The increase in ozone concentration makes ther solubility of ozone also better. This is because the more probability the ozone dissolves with the microbubbles in the water. While for ozone capacity, the greater ozone capcity per hour the smaller the dissolved ozone.

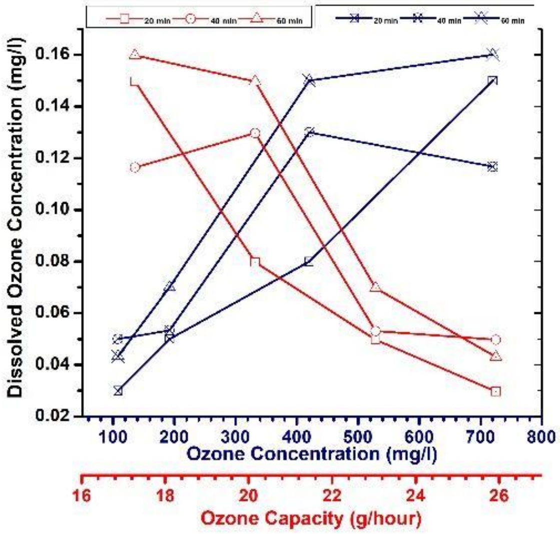
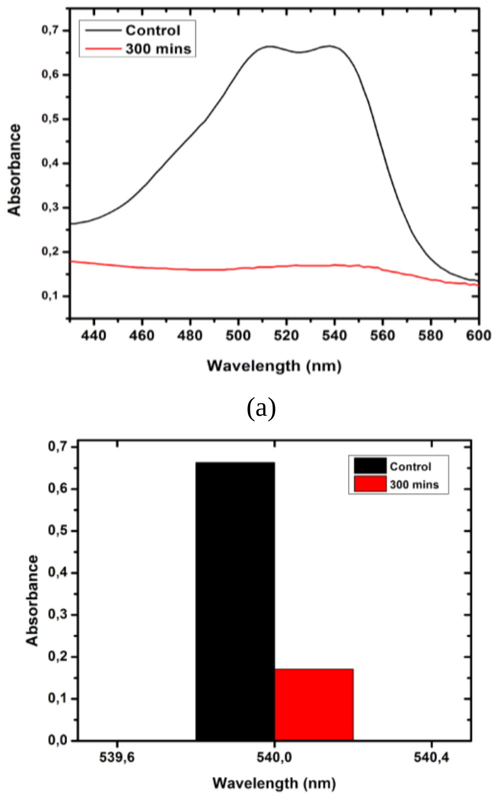


Fig. 6: Ozone Solubility Relationship with Ozone Capacity and Concentration

D. *Influence of Treatment Times on Absorbance*

Comparison of the treatment duration of the waste was analyzed specifically using the UV-VIS method which also indicated the reduction of waste parameters. The parameter of batik wastewater is directly proportional to the magnitude of the absorbance value. Fig.7 compares the absorbance values in the 400-600 nm wavelength range. This is because the characteristics of the RR 195 are also in the wavelength between 400-600 nm as shown at fig.8. The selected data is also to make it easier to analyze.



(a)

(b)

Fig. 7: (a) Absorbance batik wastewater at range 400-600 nm after 300 mins treatment and (b) bar graph of absorbance control and 300 mins after treatment at peak wavelength 540 nm.

The origin of the specific peak in the image above is from the RR 195 dye molecule and by-products (by products produced during treatment). The decrease in absorbance can be observed in Fig.7. It can be seen that the longer the treatment of waste with the ozonation process, the smaller the absorbance, which means that the longer the treatment, the less pollutant contained in the waste. The purification efficiency during treatment of 300 minutes, obtained removal efficiency value of 74.43%. Based on Fig.8, the concentration of batik wastewater before treatment (control) can be obtained, which was 0.36 ppm.

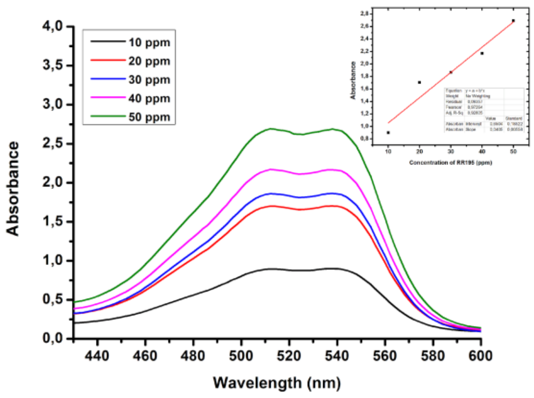


Fig. 8: Calibration curve of absorbance RR 195 solution by using UV-Vis Spectroscopy

E. Influence of Treatment Times on pH and Temperature Parameters

There are two main oxidation mechanisms in the ozonation process, the direct attack of the ozone molecule and the indirect attack of the hydroxyl radicals. The pH level of the solution affects the formation of hydroxyl radicals and determines the main oxidation mechanism during ozonation [38]. At lower pH levels or under acidic conditions, the dominant oxidation mechanism is the direct attack of ozone molecules. Whereas at higher pH levels or under alkaline conditions, hydroxyl radical attack is the predominant [39, 40].

Hydroxyl radical generation OH* is an active species that plays a role in degrading dyes. However, this study obtained the opposite result. The pH value of the waste after being treated for 300 minutes did not change using litmus paper as a pH measuring instrument, it means there is no relationship between pH and ozonation. This was due to the limitations and accuracy of the tools used. In general, the higher the pH value, the ozonation process will be more optimal [41]. However, if contaminants are more easily decomposed by direct molecular attack of ozone than by hydroxyl radical attack, ozonation will be more effective if using a small pH value [38].

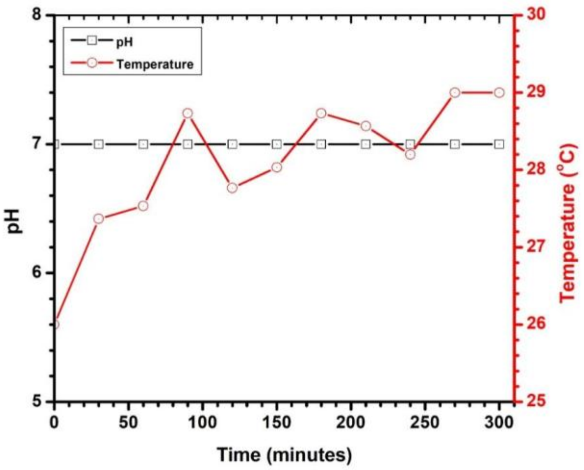


Fig. 9: The trend of pH and temperature as a function of treatment times.

The temperature of the batik wastewater increased with the length of treatment. This is because the water pump engine is hot along with the length of time the engine is running. The hot water pump engine will distribute heat to the wastewater that enters the pump as a flow. This flow occurs continuously so that the distribution of heat that is channeled from the engine to the wastewater also continues to occur so that the temperature of the wastewater increases. Initial waste temperature 26°C, increased to 29°C with treatment for 300 minutes. This did not affect the ozonation process because the temperature increase was less significant. Several studies conducted the influence of temperature on the optimization of the ozonation process by varying the temperature from 20°C to 80°C [38, 42, 43, 44].

Increasing the solubility and stability of dissolved ozone in solution for oxidizing compounds that are difficult to degrade is the key to increasing the efficiency of the ozonation process [45, 46]. Therefore, controlling the reaction temperature accordingly is an important parameter during the ozonation process [38]. The increase in temperature not only increases the thermal power of the reaction, but also affects the decay of ozone at the same time in the ozonation process. If the increase in the thermal power of the reaction is more dominant than the decay reaction, the ozonation process will be more optimal [38]. This is due to the faster decomposition of complex molecules into simpler molecules [38, 42]. If the decay reaction O_3 is more dominant than the thermal reaction power, the ozonation process will be less than optimal [47, 48]. This can be explained by the increase in temperature at O_3 is proportional to the decay reaction O_3 so that ozone will deplete with increasing temperature and tends to be stable at a certain temperature [49]. In addition, an increase in temperature will reduce mass transfer between gas and solution which causes the ozonation process to be less than optimal [46, 50].

F. Influence of Treatment Times on TDS and EC

TDS is the amount of organic and inorganic substances dissolved in the solution. The value of EC and TDS in batik wastewater is influenced by compounds in the dye such as OSO_3Na, SO_3Na, and CHCONH which causes more ions and an electric charge [51].

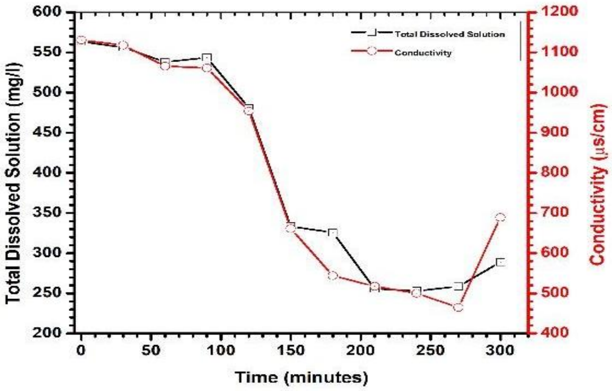


Fig. 10: The trend of TDS and conductivity as a function of treatment times

The TDS value is reduced by 565 mg/liter to 288 mg/liter and the EC value is reduced by 1130 µs/cm to 687 µs/cm after treatment using nano-microbubble ozone for 300 minutes. Seen in the Fig.6 there is an anomaly in the treatment time to 300 minutes. The TDS value increased after 270 minutes of treatment and the EC value increased for 240 minutes. This can be explained as follows, there are two important parameters that play a role in the magnitude of the conductivity value and its decrease, the chemical components used for the batik process such as sodium silicate and free carbon ions, paraffin, as well as wax chemicals attached to the fabric or dyes on the batik. These parameters directly affect the concentration of electrostatically charged substances such as salt ions in the liquid phase [52]. Nano-microbubble ozone treatment that is carried out continuously will also cause the release of hydroxyl radicals continuously, which means that this is the reason why at a certain minute the TDS and EC values will decrease, after that they will increase which in the end will tend to be stable.

G. Influence of Treatment Times on COD and BOD

Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand analysis in this research is conducted on Laboratorium Teknik Lingkungan Diponegoro University. This analysis was conducted to determine changes in organic pollutants in batik wastewater (rinsing process). Batik wastewater is treated with micro bubble ozone using 4 venturi micro bubble generators at a tme variation of up to 300 minutes.

In the control sample, the COD value was found to be 61.474 ppm. After being treated for 180 minutes, the COD value decreased to 40.421 ppm. Up to 300 minutes, the COD concentration dropped to 34.632. The COD was reduced remarkably at high ozone concentration [11]. The COD removal maybe related to the presence of organic compounds that easily oxidize and partially oxidize [53].

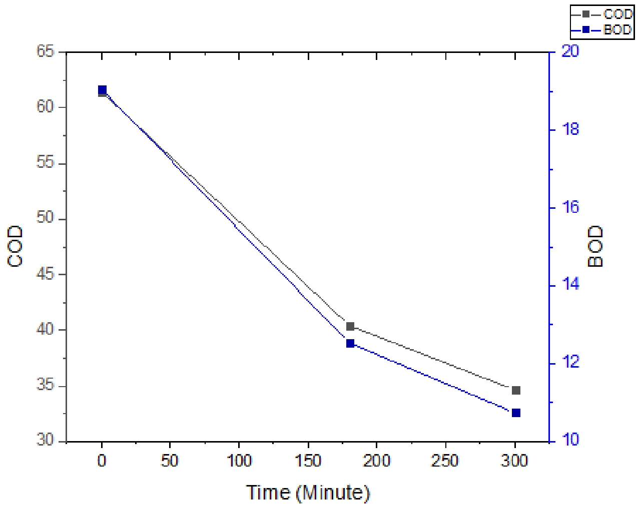


Fig. 11: COD dan BOD degradation

Based on the fig. 6 it was seen that the decrease in BOD concentration proportional with time variation. The initial BOD value of 19.057 ppm, after being treated for 180 minutes and 300 minutes, the BOD concentration decrease to 12.531 and 10.736 ppm. The decrease in BOD value can be occur because the direct oxidation reaction when ozone in the water causes insaturated bonds and will trigger the breakdown of relatively small bonds. While the reaction in an indirect way, namely, ozone utilizes hydroxyl which are he result of ozone decomposition [54].

H. Reactive Red 195 Dyes Degradation with Nano-MicroBubble Ozone

The process of degrading dye RR195 by ozone is similar to the chain degradation process and converting complex dye molecules into simpler ones. There are two possible degradation pathways for RR195 in the ozonation process proposed by Zhang *et al.* [55] shown at the Fig.12.

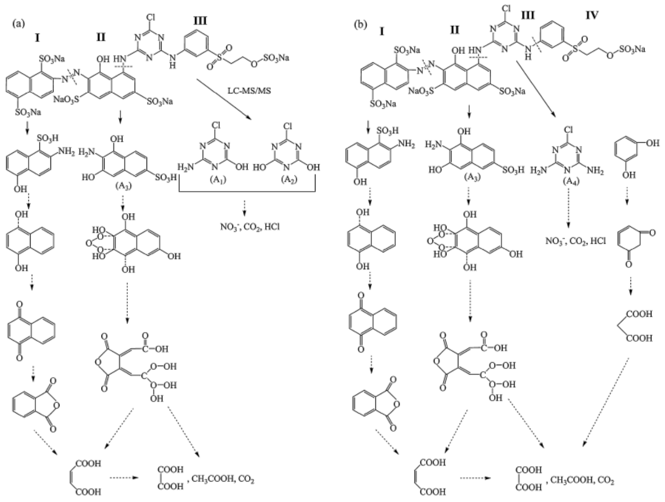


Fig. 12: Degradation pathwayRR195 with ozonation. (a) degradation pathwayNo. 1; (b) degradation pathwayNo. 2 solid lines describe degradation pathways and dashed lines indicate portions of the ozonation product that may be further degraded into smaller components [55]

The dye in dissolved RR195 is a mixture of products from two degradation pathways. Degradation pathway No. 1 in Fig.12(a) contains compounds with fragments A1, A2, and A3. While Fig.12(b) No. 2 contains compounds with A2 and A3 fragments. The bond $-N = N --N = N -$ must be cut off during the ozonation process if the efficiency is nearly 100%. The synergistic effect of ozone generates hydroxyl radicals, breaking down the RR195 dye molecule into simpler molecules. The product of the ozonation process will then be completely decomposed by CO_2 and other small organic acids if the oxidation process continues.

I. *Influence of Treatment Times on Color of Batik Wastewater*

The results of the research that have been carried out show that the longer the treatment, the lower the waste parameters. The decrease in the waste parameters is indicated by a change in color that becomes clearer.

Fig. 13: Color differences on the influence of treatment times on batik wastewater using a flow rate of 0.4 L/min

Treatment of waste for 180 minutes has shown a change in color which becomes clear as in Fig.8, then the treatment is continued for up to 300 minutes to obtain better parameter degradation.

IV. CONCLUSIONS

The application of nano-micro bubble ozone has been carried out to reduce the parameters and dyes of rinsing batik wastewater with a flow rate of 0.4 L/min. Treatment of waste for 180 minutes has shown a change in color which becomes clear then the treatment is continued for up to 300 minutes to obtain better parameter degradation. The removal efficiency is obtained by 74.43% and concentration of batik wastewater before treatment was 0.36 ppm. EC and TDS parameters experienced a significant decrease in proportion to the length of treatment, 565 mg/l to 288 mg/l for TDS, 1130 µs/cm to 687 µs/cm for EC, 61.474 mg/l to 34.632 mg/l for COD, and 19.057 mg/l to 10.736 mg/l for BOD after 300 minutes treatment. Meanwhile, the temperature increased with the length of treatment for 300 minutes, from 26°C to 29°C. This is because the longer the treatment, the hotter the pump engine which causes the system to heat up.

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