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Comparison of Ozone Pre-Treatment and Post-Treatment Hybrid with Moving Bed Biofilm Reactor in Removal of Remazol Black 5

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Abstract. The purpose of this research is to investigate wastewater treatment with ozone pre-treatment and post-treatment integrated with Moving Bed Biofilm Reactor (MBBR). Ozonation as a pre-treatment served to increase the Biochemical Oxygen Demand (BOD)/Chemical Oxygen Demand (COD) ratio while as a post-treatment to remove the remaining organic residues. Ozone doses were 1 mg/min, 4 mg/min, 20 mg/min, and 40 mg/min. In the pre-treatment, ozone produced a BOD/COD value of 0.68. The MBBR process had a COD and color removal efficiency of 68.89% and 67%, respectively. In the MBBR process without ozone pre-treatment, the efficiency of COD and color removal were 79.31% and 64.7%, respectively. The effluent treatment results from MBBR were then processed with ozone and showed the highest COD and color removal results of 76.8% and 99%, respectively. In this study, the processing with ozone as post-treatment was better than the pre-treatment.

Keywords: COD removal; Color removal; MBBR; Ozone post-treatment; Ozone pre-treatment; RB5

1. Introduction

Dyes are organic compounds having the capability to impart their colors to materials such as textiles and fabrics. To date, the textile industry still contributes significantly to the economic growth of Indonesia (Salamah et al., 2019). This is reflected in the growing demand for dyes in the production and industrial processes. During the coloring process, 10–15% of the dyes are discharged with effluent waste. The high use of coloring agents in certain industrial activities has an unfavorable impact on the increasing amount of pollutants in wastewater. This happens in particular small and medium scale textile

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industries. It is a common practice to directly discharge the textile industry wastewater into a stream or the environment (Apritama et al., 2020) and this has had a negative environmental impact (Sharfan et al., 2018).

Dyes used for textiles are frequently natural extracts or synthetic (artificial) dyes. Natural dyes, in comparison to synthetic dyes, are more favorable since they are less carcinogenic (Manikprabhu and Lingappa, 2013). However, the ease in obtaining synthetic dyes and their affordable prices maintains their demand at high levels in the global market. Synthetic dyes in textiles are aromatic hydrocarbon derivatives such as benzene, toluene, naphthalene, and anthracene (Fitriana and Adriani, 2019). Synthetic dyes possess greater stability than natural dyes. Based on the color indexes, the largest and most versatile dyes belong to the azo group, and most of them are reactive. One of the extensively used reactive dyes in textiles, due to its easy application, is Reactive Black 5 or Remazol Black 5 (RB5) (Pratiwi et al., 2018). Based on chemical elements, RB5 contains azo chromophore groups, used as black dyes in textiles. RB5 has the molecular formula $C_{26}H_{21}N_5Na_4O_{19}S_6$ and a molecular weight of 991.82 g/mol (Bazrafshan et al., 2015; Samadi et al., 2015).

The wastewater treatment in the textile industry can be achieved by chemical, physical, and biological processes or a combination of the three. The biological wastewater treatment is considered one of the simplest and affordable treatment alternatives. This biological wastewater treatment can be integrated with a preliminary treatment such as ozonation (Pratiwi et al., 2018; Suryawan et al., 2019). Ozonation can degrade the color RB5 and, consequently, generate aromatic and aliphatic acids convert it into CO_2 and H_2O. On the contrary, when ozone is used separately, the optimum result of processing cannot be accomplished. Thus, the combination of ozone treatment with biological treatment such as MBBR is highly recommended. Textile wastewater processing with activated sludge requires 2-5 days of hydraulic retention time (Haddad et al., 2018). This technology must maintain the amount of dissolved oxygen with aeration (Anaokar et al., 2018; Suryawan et al., 2020a). The activated sludge process is very sensitive to shock loads, especially the organic loading rate (Manai et al., 2017) and the salinity (Mirbolooki et al., 2017). Advanced treatment with filtration such as microfiltration, ultrafiltration, nanofiltration, and reverse osmosis in textile wastewater treatment can cause dye molecules to clog the membrane pores (Kumar et al., 2019).

MBBR has emerged as a promising technology since it has an anti-shock capability and high biomass density (Gong, 2016). In addition, MBBR has a high biofilm and long sludge retention time (Jabri et al., 2019). Borkar et al. (2013) stated that MBBR could potentially overcome the challenges in industrial wastewater by improving the efficiency of both conventional treatment systems and removing nutrients, reducing sludge production due to long biomass retention time, minimizing process complexity such as backwashing and maintenance, and providing affordable cycle. The application of ozone can be utilized as pre- or post-treatment. Therefore, the purpose of this research is to investigate the efficacy of the ozone technology as pre-treatment and post-treatment in the MBBR technology.

2. Methods

2.1. Materials

Ozone at dosages of 1 mg/min, 4 mg/min, 20 mg/min, and 40 mg/min was introduced into the pre-treatment or post-treatment ozone reactor. Kalium Iodida (KI) solution was prepared in the laboratory to absorb excess ozone gas and safety so as not to cause harm (Septiariva et al., 2020). The volume of the flow-batch reactor of the ozone and the MBBR reactor was 2 L each (see Figures 1–2). The wastewater used in this research was artificial wastewater with the addition of one gram of starch as organic material, predominantly adopted in the Indonesian textile industry. The ozone dose was determined by Standard Method No. 2350 E and by the iodometric method.

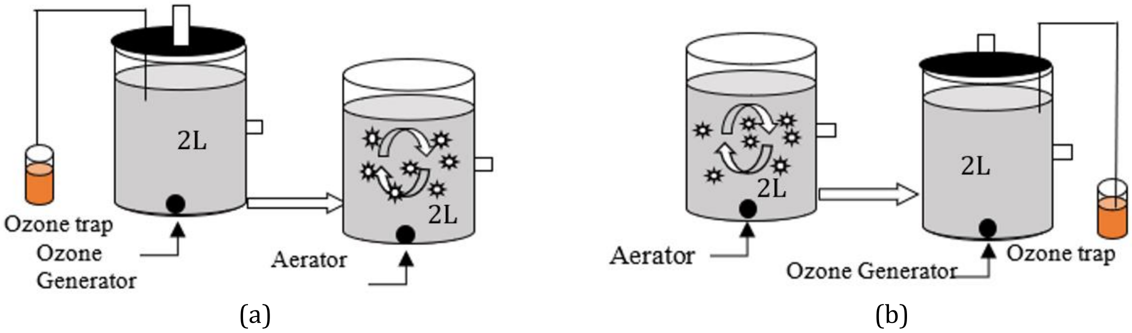


Figure 1 RB5 Processing flow chart with ozonation as: (a) Pre-treatment in the MBBR unit; and (b) A post-treatment in the MBBR unit

Figure 1 shows the RB5 wastewater pre-treated with ozone before MBBR processing and post-treated after MBBR processing. The media employed in the MBBR unit was the Kaldness k1 (Figure 2a). Detention time to form the attached growth of microorganisms was two weeks. The bacteria to form the attached growth were derived from activated sludge seeded in the laboratory (Figure 2b).

2.2. Data Collection

Sampling during the ozone treatment process was carried out in a period of 2 h, where the two-hour detention time is the optimum time for said process (Suryawan et al., 2020a). As for the MBBR process, the sampling was carried out for 1 h until the COD results were saturated. Sample testing was carried out without preserving the sample. Standard Methods No. 5220 and 5210 were used to estimate COD and BOD. A UV-vis spectrophotometer was used at 595 nm to quantify color concentration. Standard color solutions of different concentrations were prepared for the standard peak curve.

2.3. Calculation of Biological Treatment Parameters

The value of μ is one indicator of the biodegradability level of the wastewater treatment. In a short period (dt), the increase in the amount of biomass (dx) is proportional to the amount of available biomass (X) (Equation 1). A low value of μ indicates slow microorganism growth.

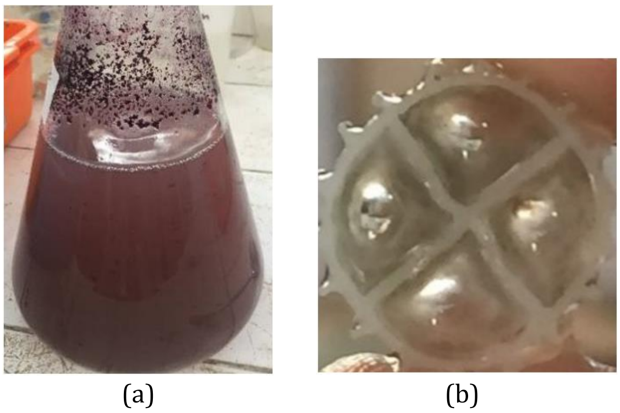


Figure 2 (a) Bacterial seed growing Kaldness k1; and (b) the Kaldness k1 medium for the MBBR reactor

$$\frac{dx}{dt} = \mu\dot{X} \tag{1}$$

$$ln\left(\frac{Xn}{Xo}\right) = \mu t \quad (2)$$

where dx/dt is the population growth rate, μ is the specific growth rate, and X is the growth rate of the unity of the amount of biomass. The μ value can be determined using Equation 2. The number of biomass cells increases exponentially, and the specific growth rate equation is obtained as follows: Xn is the biomass concentration on an nth day, Xo *is* the biomass concentration on day 0, and t is the nth time.

2.4. Calculation of Substrate Degradation Rate (q) and Yield (Y)

The specific speed for the substrate removal follows the first-order reaction (Equation 3). By plotting the value of ln(Co-C) versus time, the value of the substrate degradation rate (q) was obtained. The decrease in COD indicates that optimum conditions could be achieved for microorganisms to degrade or remove the organic compounds in wastewater. Yield (Y) is the comparison between the coefficient of the biomass amount and the mass of the removed substrate in biological treatment. Cell production per substrate utilization increases along with the level of Mixed Liquor Volatile Suspended Solids (MLVSS). In this study, the substrate was analyzed based on COD concentration and color (C) as the substrate concentration in mg/L (Equation 3).

$$\frac{dC}{dt} = q\, C^n \quad (3)$$

3. Results and Discussion

3.1. MBBR with Ozone Pre-Treatment

The objective of this experiment was to study the effect of preliminary treatment on influent wastewater. The initial RB5 dye concentration was set at 100 mg/L and the starch concentration of 1 g/L was added. At initial concentration, the results for RB5 color measurements, BOD concentration, COD concentration, and BOD/COD ratio were 100±8.9 mg/L, 41±5.5 mg/L, 153.4±41.2 mg/L, and 0.26 respectively as presented in Figure 3a. Ozonation at the dose of 4 mg/min gave a maximum value of 0.68 for BOD/COD with a two-hour detention time. The addition of starch at 1 gr/L shifted the COD value to 1192±121.7 mg/L and the BOD/COD value to 0.23 (Figure 3b).

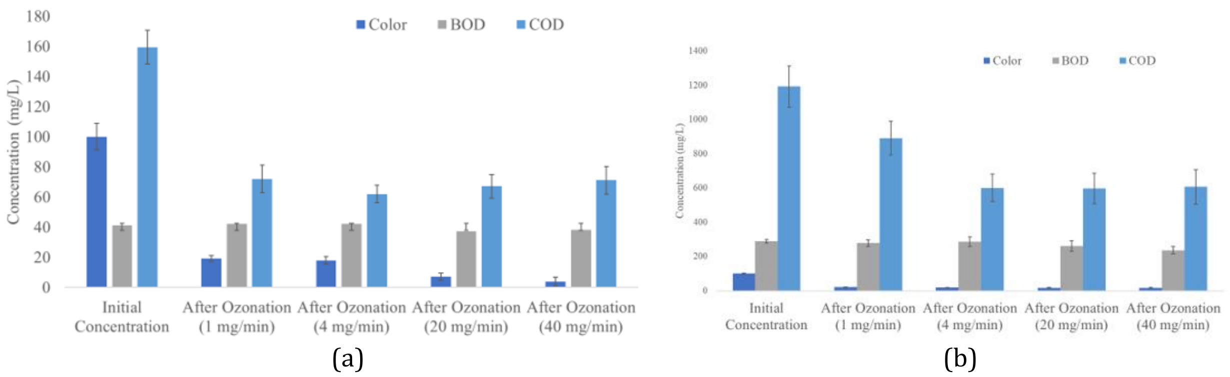
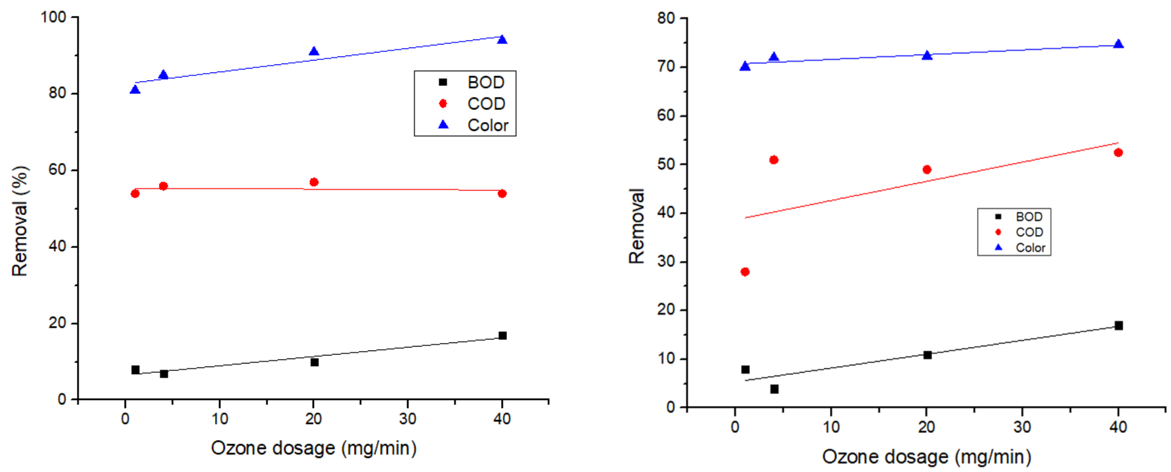


Figure 3 The results of pollutant parameters in an RB5 solution (100 mg/L) with a detention time of 120 minutes: (a) after the addition of starch (1 g/L); and (b) without starch addition

The maximum increase in BOD/COD of 0.48 (Figure 3c) was attained after ozone pre-treatment at a dose of 4 mg/min. Preliminary treatment with ozone provides some advantages for the removal of toxicity in dyes, destruction of aromatic and aliphatic acids , organic material, and enhancing biodegradability (Karamah et al., 2019; Suryawan et al., 2020a). A detention time of 120 minutes and an ozone dose of 4 mg/min yielded 91.6±0.8%

of RB5 color removal efficiency without starch addition. On the contrary, the removal efficiency after adding the starch was only 75.2±0.7%. This suggests that the bond between the dye and starch hinders the oxidation of the dye. COD can be removed at a percentage of 46.41±4%, while with the addition of starch the COD efficiency became 28.6±6%, with an initial color concentration of 100 mg/L. Moreover, the addition of startch and RB5 do not always have a positive correlation with the drecreasion of COD removal, because the bond between RB5 and starch could obstruct ozone oxidation.

In the experiment done by Bilińska et al., (2016) with an initial color concentration of 125 mg/L, the COD removal increased from 80% to 90% in 60 minutes. Figure 4 shows the correlation between ozone dosing and the percentage of RB5 dye removal. The pollutant parameters were color, BOD, and COD values. In color removal, there is a high correlation between the ozone dose and the percentage of color removal in RB5 solution without and with starch addition. The correlation coefficient for this parameter in the RB5 solution is 0.8948, while with the starch addition it is 0.8625 (Table 2). In COD removal, RB5 solution and starch addition had a low correlation coefficient, 0.0025 and 0.3718 refer to the removal COD and colour parameter (Table 2).



(a) (b)

Figure 4 Liniar correlation between the ozone doses and the removal of pollutant parameter: (a) in a 100 mg/L RB5 solution with a detention time of 120 minutes; and (b) in a 100 mg/L RB5 solution and the addition of starch at 1 g/L with a detention time of 120 minutes

The correlation value between COD removal and ozone dose was low because the organic matter left in the wastewater became resistant to chemical oxidation by ozone. BOD parameter demonstrated a strong correlation with the ozone dosage both with and without starch addition, as shown in Figure 4. Gifford et al. (2018) also mentioned that higher ozone doses would consequently increase the biodegradability of organic substances. Our finding is in line with Ebrahim et al. (2018), where it is reported that strong correlations were obtained for color and BOD parameters on pharmaceutical removal. However, the color removal was higher than the COD removal because of the changed form of the organic compound in the RB5 ozonation treatment. The increase in BOD/COD shows that the higher the dose of ozone the more susceptible the organic substances become to microorganisms. This also reinforces the finding that the RB5 in wastewater is not completely oxidized but is converted into simpler organic compounds so that BOD/COD increases. The absence of a high correlation between the ozone doses and COD might be due to the incomplete degradation of organic compounds to CO_2 and H_2O. Intermediate short-chain organic compounds might be formed and subsequently lead to higher BOD. Besides, increasing the ozone dose could increase COD because the refractory organics accumulated in the eluate (Li et al., 2020).

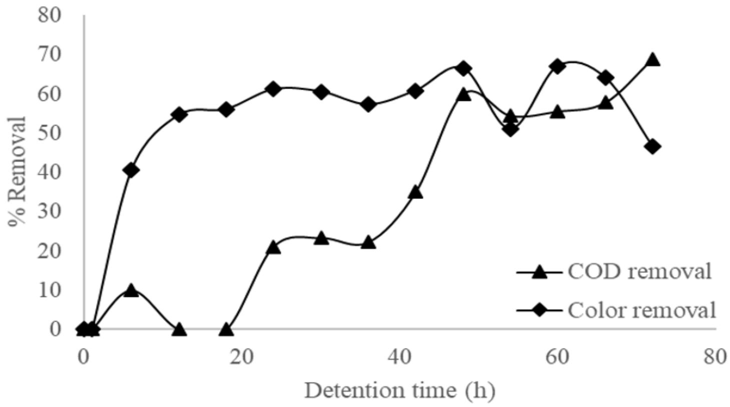


Figure 5 The effect of ozone addition at 4 mg/min within the MBBR unit to the decreasion of COD and colour

Table 1 Biological parameters for treating the effluence of ozone at 4 mg/min in the MBBR unit

Parameters	MBBR with Ozonation Pre-treatment	MBBR without Ozonation Pre-treatment
μ	0.0036 d^{-1}	0.0075 d^{-1}
q COD	0.0177 d^{-1}	0.0163 d^{-1}
q Color	0.0048 d^{-1}	0.004 d^{-1}
Y COD	0.5300 mg MLVSS/mg COD removed	1.11 mg MLVSS/mg COD removed
COD removal	68.90%	79.31%
Color removal	67%	64.70%
COD effluent	89.6 mg/L	194.7
COD quality standard	150 mg/L*, 100 mg/L**	

*Indonesia (Kementerian Lingkungan Hidup Republik Indonesia, 2014), **Taiwan (ZDHC, 2016)

The effluence of ozonation at a dose of 4 mg/min in the RB5 solution with starch was processed in the MBBR unit. The process was performed by considering the steady-state approach in the COD and color removal. The duration to produce a steady state was 48 to 72 hours (Figure 5). The highest removal of COD and color obtained within the specified time interval was 68.89% and 67% respectively (Table 1). According to Equations 1 and 3, the value of biological treatment parameters was estimated and is presented in Table 1. The specific growth rate in this treatment had the value of 0.0036 d^{-1} (Table 1). The specific growth rate in the MBBR unit (0.0315 d^{-1}) showed a lower value as compared to the integration of ozone pre-treatment with activated sludge in textile waste (0.1379 d^{-1}) (Suryawan et al., 2019). This shows that the treatment with suspended media produced more sludge as compared to the attached media, namely the MBBR unit. The yield from the textile waste treatment with ozone had the value of 0.53 mg MLVSS per mg COD removed, using a reactor volume of 0.61 m^3. Despite the low value obtained as compared to international regulations, the results of wastewater treatment with the integration of ozone and the MBBR unit have met the effluence quality standards for textile wastewater based on Indonesian regulations.

3.2. *MBBR with Ozone, Post-Treatment*

The ozone processing as pretreatment used to obstruct organic matter into biodegradable organic. This also happened in previous research, which stated that chemical or physical processes could not provide selective removal, they were expensive, and not environmentally friendly (Iswanto et al., 2019). Ozonation as a post-treatment after the MBBR processing aims to remove recalcitrant organic residues that cannot be degraded in the ozonation as a pre-treatment and the process of MBBR. Figure 6 shows that the COD removal efficiency is more stable than the color removal efficiency. At a period of 48 to 72 hours, the maximum COD removal efficiency reached 79.31%. On the contrary, the percentage of color removal increased within 48 hours and then decreased until reaching the 72-hours window. The maximum efficiency achieved for the detention time of 48 hours for color removal was 64.7%. Color removal in biological treatment depends on the mechanism of desorption and adsorption on the surface of the MBBR media. Gong (2016) stated that the initial concentration of COD at 47 mg/L in an MBBR unit resulted in 94.3% COD removal.

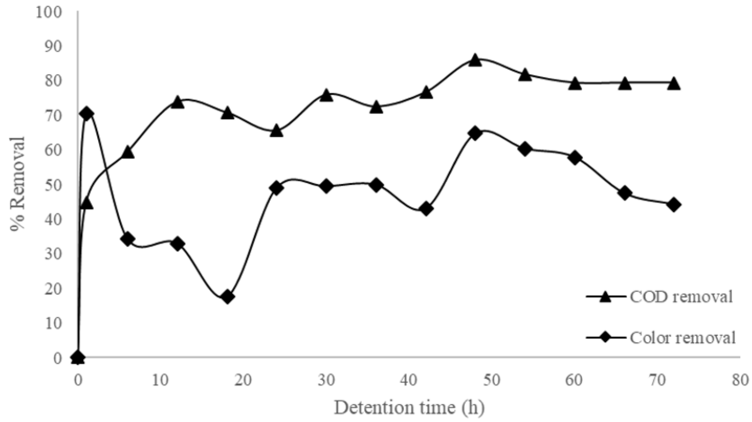


Figure 6 Parameters of pollutant removal with RB5 dyes at 100 mg/L and starch solution at 1 gr/L with an MBBR unit

Table 2 Correlation of the ozone dosing with the removal of pollutant parameters in ozonation treatment

Treatment	R^2 Correlation		
	BOD	COD	Color
100 mg/L RB5 solution	0.9364	0.0025	0.8948
100 mg/L RB5 solution and the addition of 1 g/L starch	0.8667	0.3718	0.8625
MBBR effluence	No measurement	0.9747	0.6282

In the absence of ozone pre-treatment, specific growth rates for microorganisms had a value of 0.0075 d^{-1}, which is higher than the MBBR treatment with ozone pre-treatment (Table 1). In the present study, COD yield had a value of 1.1 mg MLVSS/mg COD. The ozonation treatment with different doses of MBBR effluence with 72-hours detention time is presented in Figure 7a. The results of processing with pre-treatment and post-treatment show that they could properly remove the organic matter such as color and COD so that it met quality standards. The determination to the right detention time by looking at the colour changing during ozonation. In post-treatment processing, the improvement of the oxidation results cannot be separated from the simple changes of organic compounds in wastewater carried out by microorganisms.

The results imply that the ozonation processing was effective in removing color and COD. More than 99% of color removal could be achieved with doses of 20 mg/min and 40 mg/min. The obtained percentages of COD removal were between 67.2% and 76.8%, which meets the quality standards for wastewater in Indonesia. The high efficiency of color due to the cleavage of the azo C-bond reaction occurs only in the chemical physics reaction with ozonation. In MBBR, it tends to reduce organic bonds that are not azo. The still high levels of COD in RB5 are due to further oxidation of the azo ring which produces low molecular weight organic acids (for example, acetic and oxalic acid), carbon dioxide, and water (Zheng et al., 2016).

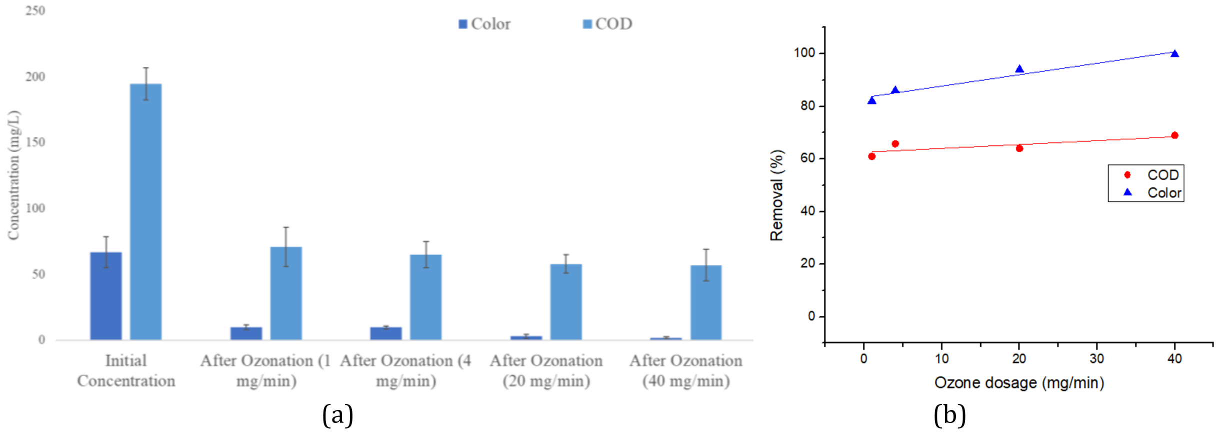


Figure 7 (a) Results of pollutant parameter measurements on the MBBR effluent unit with a detention time of 120 minutes; (b) Linear correlation of ozone dosing with the removal of pollutant parameters in the MBBR effluent unit with a detention time of 120 minutes

It is observed that the post-treatment approach offered a more satisfying result than pre-treatment. Similar findings of ozonation as post-treatment for wastewater treatment have been reported (Morali et al., 2016; Suryawan et al., 2020b). In a study by Punzi et al. (2015), the integration of treatment with an anaerobic biofilm reactor followed by ozonation detected no mutagenicity. Hence, ozone treatment as a follow-up treatment does not have adverse effects and can be discharged into a body of water. The ozone dosage correlation shows a positive value on color removal and COD (Figure 7b) for ozone post-treatment.

4. Conclusions

The results of color, COD, and BOD removal from wastewater subjected to pre-treatment and post-treatment with ozone meet the textile industry wastewater quality standards. However, the treatment with ozone post-treatment provides better efficiency than the pre-treatment. More than 99% of color removal could be achieved with each ozone dose. The concentration of COD in effluence reached 89.6 mg/L and 53.44 mg/L with ozone pre-treatment and post-treatment, respectively. Even though it has met the quality standards of COD, the COD content in RB5 wastewater did not reach the mineralization stage. A color efficiency of up to 99% was used as the basis for enhancing the value of textile waste, which gives an unusual color to the water bodies. A pilot study is needed to test the effectiveness of the technology for both the ozone pre-treatment and post-treatment along with the MBBR process.

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