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Removal of Methylene Blue Intensified Ozonation Using a High-Performance Rotating Reactor

Phuong Thao Nguyen*, Minh Viet Trinh, Thi Doan Pham, Van Tuyen Trinh, Thi Thom Dang, Linh Phuong Phung, Tuan Minh Nguyen
Institute of Environment Technology, Vietnam Academy of Science and Technology, Ha Noi, Vietnam
**Corresponding author email: phuongthaobk56@gmail.com*

Abstract

The advanced oxidation processes (AOPs) were commonly used in textile dyeing wastewater treatment due to its ability to rapidly degrade dyed compounds. However, its disadvantage is the limited dispersion of ozone in wastewater. To minimize the disadvantage, it is necessary to improve the gas-liquid phase contact to maximize the dispersion of ozone in the wastewater body. Therefore, this study investigated the methylene blue (MB) treatment efficiency in textile dyeing wastewater by performing the ozonation process using a high-performance rotating reactor (HP2R) and compared it with the conventional system. The effect of basic operation parameters such as initial pH, initial methylene blue concentration (C_o), rotational speed (ω), and the liquid flow rate (Q_L) on the decolorization efficiency (η) was evaluated. The results showed that the η of the ozonation using the HP2R reached 98% (pH 8) after only 5 minutes, which was 1.8 times higher than that (54%) of conventional ozonation at the identical period. The decolorization efficiency increased with increasing rotational speed (from 82.0 to 92.5% as ω increased from 30 to 1200 rpm), decreasing liquid flow rate (from 98.0 to 74.9% as Q_L increased from 0.1 to 0.4 l/min) and decreasing initial concentration of MB (from 98.0 to 88.5% as C_o increased from 50 to 200 mg/l). These results show that the combined ozonation in the high-gravity technology can be widely applied to various dyeing wastewater treatment processes in general.

Keywords: Rotating packed bed, ozonation, decolorization, methylene blue, textile dyeing wastewater, HP2R.

1. Introduction

Organic pollutants are extensively present in textile dyeing wastewater. This pollutant has a stable and complicated color molecular structure. The substances are hazardous and challenging to be treated using conventional physical, chemical, and biological techniques. Methylene blue (MB) is one of the most popular dyes used in the textile industry. The color of wastewater interferes with the absorption of oxygen and sunlight, which is detrimental to the respiration and growth of aquatic organisms, and adversely affects the ability of microorganisms in wastewater. For humans, some dyes can potentially cause skin, respiratory, lung, and even cancer [1].

There are many methods to treat methylene blue in textile dyeing wastewater. The most common physical method in textile dyeing wastewater treatment is adsorption, which has the advantages of simplicity in design and operation, and low cost [2]. However, these methods are rarely chosen due to the short material life cycle and vulnerable to factors such as temperature, chemical oxygen demand (COD), biological oxygen demand (BOD), pH, and heavy metals [2]. Biological methods are often used in wastewater treatment because of their high applicability and low cost [2]. However, the application of biological treatment methods will have to be accompanied by steps of 1st and 2nd physicochemical treatments to ensure the output wastewater quality. Advanced oxidation processes (AOPs) have been demonstrated to be effective in reducing organic compounds in wastewater. AOPs are a collection of strong technologies that can be used to cure water, including ultraviolet (UV) radiation, ozone (O_3), hydrogen peroxide (H_2O_2), and oxygen (O_2). The Fenton process, ozonation, catalytic wet air oxidation, electrochemical oxidation, or even a mixture of several of these AOPs are some of the more prevalent ones [3]. Ozonation is regarded as one of the most efficient methylene blue therapy techniques among AOPs. In the decoloration of dye wastewater, ozonation is preferred thanks to its high color treatment efficiency with simplicity in operation. However, ozonation is significantly affected by the gas-liquid mass transfer rate due to the low solubility of ozone in water, which makes the amount of ·OH was limited. Besides, the O_3 residue caused the emission of greenhouse gases to the environment.

Ramshaw and Mallision had the idea to create a rotating packed bed that allows liquid to flow and distribute in the contact cushion with centrifugal acceleration, which is 10 - 100 times the gravity to overcome the disadvantages of traditional gas-liquid

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contact equipment [4]. The rotating bed is connected to the rotor, which rotates from 300 to 1800 rpm, creating a centrifugal acceleration that is from 100 to 1000 times greater than the gravity [5]. With the high centrifugal, mass transfer efficiency is greatly increased by being able to operate at high gas - to - liquid flow ratio (Q_G/Q_L) ratios without the risk of fluid clogging. Under the high centrifugal acceleration, the Q_L in the rotating bed surface becomes thin films that increase the gas-liquid phases contact surface area, increasing the mass transfer efficiency compared to packed columns [6]. The equipment has been widely applied as stripping in wastewater treatment, distillation, and absorption in emission treatment [7-10]

There are many studies have been conducted to investigate the treatment efficiency of rotating packed beds in wastewater treatment. The effectiveness of rotating packed beds was studied through the removal of Volatitle Organic Compounds (VOCs) stripping from groundwater [8], stripping ethanol [9], and stripping ammonia [10]. In a pilot-scaled Rotating packed bed (RPB), Yuan *et al*. (2016) showed that a continuous flow of ammonia stripping from synthetic wastewater (Q_G of 9000 L/min, Q_L of 5 L/min, and rotational speed of 1000 rpm) exhibited greater stripping efficiency (95%) with a smaller packed depth than traditional packed column [7]. Trinh *et al.* (2022) reported that the ammonia stripping efficiency of using a high-performance rotating reactor (HP2R) could achieve excellent efficiency of 92.7% in 2 L of synthetic ammonia wastewater within 20 minutes [11].

With the advantage of significantly improving mass transfer efficiency in the liquid-gas phase contact of HP2R, the study of methylene blue treatment by combined ozonation and HP2R was carried out. In this study, the effectiveness of the ozonation process combined with the HP2R was evaluated through the change in the concentration of methylene blue when changing the working parameters of wastewater (pH, initial methylene blue concentration) and working parameters of HP2R (Q_L and rotational speed). The performance of the ozonation using HP2R was also compared with the traditional ozonation process for methylene blue treatment. The results of the experiment provide further insight into the feasibility of applying HP2R equipment in the ozonation process for wastewater treatment.

2. Materials and Methods

2.1. Material and Equipment

The synthetic dye wastewater was prepared from the dilution of stock MB solution (Merck, Germany). The ozone generator (NEXT PLUS 10, Germany) used in this study was used as a source of O_3 with a maximum ozone capacity of 10 g/h.

Table 1. The technical parameters of HP2R equipment

Parameters	Unit	Value
Packed bed inner radius (r_i)	m	0.024
Packed bed outer radius (r_o)	m	0.083
Packed bed average radius (r_{avg})	m	0.054
Height of packed bed (Z_B)	m	0.039
Packed bed volume (V_B)	m^3	7.74×10^{-4}
Packing material		Stainless-steel wire mesh

The HP2R used in this study was made of SUS 304 stainless steel, including liquid distribution holes, a static housing, a rotating packed bed, and packing material of stainless steel wire mesh. The parameters are described in Table 1.

2.2. Experimental Procedure

2.2.1. Expriment of conventional ozonation

Fig. 1 depicts schematically the experimental setup in this study. 2 L of MB wastewater was added to the 5-L glass reaction tank. Ozone was generated and controlled by an oxygen flowmeter (0.05-0.2 L/min) and soaked into the tank. The outlet sample was collected at the discharge valve of the vessel. The output ozone was bubbled to the 2% KI solution to remove excess ozone before being discharged into the environment.

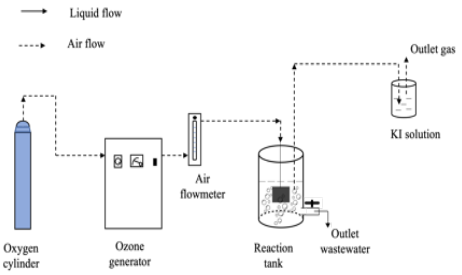

Fig. 1. Conventional ozonation experimental setup

In this study, the experiments of conventional ozonation were conducted at various conditons of pH (2-10) and liquid flow rate (Q_L of 50 to 100 mg/L), ozone concentration was 0.06 g/L (Q_G = 0.5 L/min).

The output MB concentration was collected and analyzed after 5; 10; 20; 30 minutes. The triplicate test was performed in every set of experiment.

2.2.2. Experiment of combined ozonation with HP2R

The experimental setup of ozonation combined with HP2R is presented in Fig. 2. In this experiment, the reaction tank was replaced by the HP2R equipment to derive the dispersion of O_3 and MB wastewater. Instead of filling the MB wastewater in a container, a peristaltic pump (EHN-C36VH4R, Iwaki, Japan) was used to continuously feed the liquid into the HP2R equipment. Under various stage of rotational speed, the O_3 gas was counter-currently contacted with the fed wastewater. The output gas was bubbled to the reactor containing 2% KI solution to absorb excess ozone before being discharged into the environment.

To compare the performance in MB treatment by ozonation method combined with HP2R with previous studies, the effect of the operating parameters such as pH (from 2 to 10); Q_G (from 0.5 to 1 L/min); Q_L (from 0.1 to 0.4 L/min), and rotational speed (from 300 to 1200 rpm) were comprehensively investigated. The experiments were carried out with parameter ranges appropriate to the allowed working parameters of the equipment because the laboratory's size still places a restriction on the equipment's capacity. For each experimental condition, sampling and analysis were performed at the end of the experiment. The triplicate test was performed in every set of experiment.

The effect of initial solution pH (2-10) and the effect of MB concentration (50-200 mg/L) were conducted at the constant Q_G of 0.06 g/L; Q_L of 0.1 L/min. The effect of the rotational speed was conducted at Q_G of 0.5 L/min, C_o of 100 mg/L while the ω varied from 300 to 1200 rpm. For the experimental of the effect of Q_L, Q_L of varied from 0.1 - 0.4 L/min, the two Q_G were 0.5 and 1 L/min, and the rotational speed ranged from 300 to 1200 rpm. The reaction time was determined as 5 minutes which equals the total time for a single circular of wastewater fed into the HP2R equipment.

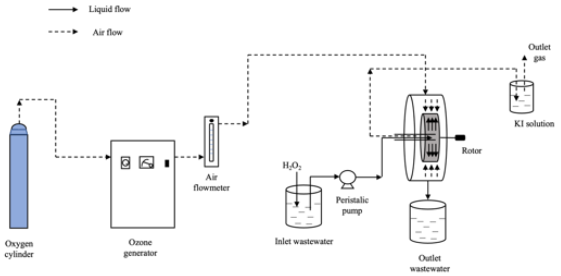

Fig. 2. Schematic diagram of the combined ozonation with HP2R experimental setup

2.2.3. Decolorization circulation experiment

The circulation experiment was performed with the MB concentration of 100 mg/L; the rotational speed ω = 600 rpm, the Q_L = 0.4 L/min, and the two Q_G were 0.5 L/min and 1 L/min. These conditions are carried out for a conventional ozone system. The effectiveness of MB treatment was compared using the conventional ozone and combined ozonation with HP2R system under the same conditions.

2.3. Analytical Methods and Calculation

The pH of the solution is measured by the HI 5222 Hanna, USA. The MB concentration before and after was measured by UV-Vis Double Beam PC Scanning Auto Cell (UVD-3200, Labomed Inc, United States). The MB calibration curve was constructed from 01; 0.2; 0.5; 1; 2; 5 mg/l at λ = 665 nm [12]. The standard curve equation has the form:

$$y = 5.1876x - 0.1597 \ (R^2 = 0.9976) \quad (1)$$

The efficiency of the process was calculated based on the change of MB concentration before and after the reaction according to the following:

$$\eta = \frac{C_o - C_t}{C_o} \times 100\% \ (\%) \quad (2)$$

where C_o and C_t are the input and output concentrations, respectively, of methylene blue in the solution (mg/L)

The ozone concentration was determined using the iodometric titration method. Ozone was soaked in 50 mL of 0.2M KI solution for 2 minutes, ozone reacted and reduced the concentration of KI in solution. The output liquid was adjusted to pH from 1-2 with H_2SO_4 and titrated the excess KI with 0,4 M $Na_2S_2O_3$ solution until the solution turns from yellow to colorless [12, 13]. The mechanism of the titration was described *as follows*:

$$O_3 + 2KI + H_2O \rightarrow I_2 + 2KOH + O_2 \quad (3)$$

$$3O_3 + KI \rightarrow KIO_3 + 3O_2 \quad (4)$$

$$KIO_3 + 5KI + 3H_2SO_4 \rightarrow 3K_2SO_4 + 3I_2 + 3H_2O \quad (5)$$

$$I_2 + 2Na_2S_2O_3 \rightarrow Na_2S_4O_6 + 2NaI \quad (6)$$

Ozone concentration was then calculated as follows:

$$C = \frac{24 \times V_{Na2S2O3} \times M_{Na2S2O3}}{1000 \times Q_G \times t} (\text{g/L}) \quad (7)$$

where $V_{Na2S2O3}$ was titrant volume (ml); $M_{Na2S2O3}$ was $Na_2S_2O_3$ concentration (mol/L); Q_G was O_3 flow rate (L/min); t was the soaking time (min)

3. Results and Discussion

3.1. Influence of Initial Solution pH

Fig. 3 shows the change in methylene blue treatment efficiency when applying conventional ozonation and ozonation combined with HP2R.

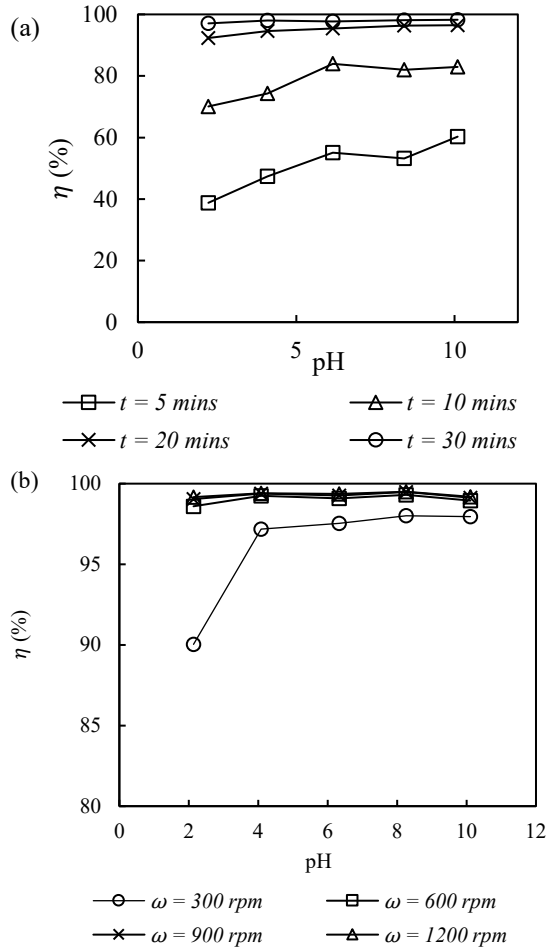

Fig. 3. Effect of pH on decolorization efficiency (a) conventional ozonation, (b) ozonation combine HP2R

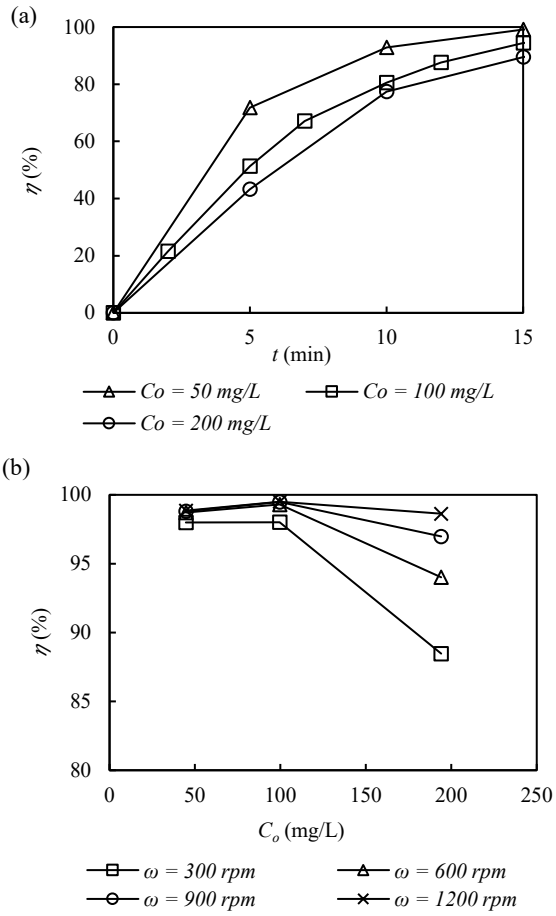

Fig. 4. The influence of the initial MB concentration on the MB treatment efficiency of the (a) conventional ozonation and (b) ozonation combined HP2R

In the first 5 minutes of the reaction, the MB treatment efficiency of the ozonation process increased from 38.8 to 60.3% when the pH increased from 2 to 10, the process was stable at 20 minutes (η increased from 92.3 to 94.2%) (Fig. 3a). The rate of decolorization increased with increasing pH, this increasing trend was consistent with the previous study by Konsowa *et al*. and López-López A *et al*. [14, 15]. For the HP2R combined ozonation (Fig. 3b), at constant wastewater properties, the decolorization efficiency increased significantly from 90.0 to 98.0% when the pH increased from 2 to 10. When increasing the rotational speed from 600 to 1200 rpm, the increasing rate of decolorization efficiency slowed down and almost remained steady, ranging from 98.0 to 99.5%. pH is one of the crucial factors that regulates the quality of the water because pH can determine the rate of reaction of substances in water [12], but at this time, the changing pH less important role due to the presence of a large amount of hydroxyl radicals, which makes the decolorization efficiency faster and stronger.

Compared to the same reaction time, the reaction rate of the HP2R combined ozonation was 3 times faster than that of the conventional ozonation system. This indicated the effectiveness of the combination of the ozonation and HP2R in the decolorization of MB. The centrifugation process improved the mass transfer efficiency from the gas to the liquid phase, which overcomes the shortcomings of conventional ozonation and brings more expectations in the decolorization of textile dyeing wastewater. This was consistent with Shuo *et al*. (2013) suggested that the dissolved ozone concentration in the ozonation process depended mainly on the gas-liquid interference and the amount of input ozone gas [16].

3.2. Influence of Initial Methylene Blue Concentration

The influence of the initial MB concentration on the MB processing efficiency of the model system is indicated in Fig. 4.

Fig. 4 shows that the decolorization efficiency of the combined ozonation and HP2R was higher than conventional ozonation. At the initial MB

concentration of 50 mg/L, the decolorization efficiency increased from 71.7% (conventional ozonation) to 97.7% (ozonation combined HP2R with ω = 300 rpm), the increase was similar to the initial methylene blue concentrations of 100 mg/l and 200 mg/l for the first 5 min of the reaction. In the conventional ozonation, the decolorization efficiency gradually increased and stabilized ($\eta > 90\%$) at the 15th minute. For the ozonation combined HP2R, the decolorization efficiency decreased linearly with increasing initial MB concentration for all rotational speeds. When the initial MB concentration was increased from 50 to 200 mg/L, the decolorization efficiency decreased from 97.9% to 88.5% at 300 rpm. With the constant ozone concentration, the gradual increase in methylene concentration leads to ozone deficiency, which reduces MB decomposition efficiency.

This conclusion was similar to the study of Xu *et al*. (2023) in the treatment of Basic Red 46 [17] and Chia *et al*. (2003) in the treatment of methylene blue [18]. However, when increasing the rotational speed by 4 times (ω = 1200 rpm) the efficiency remained unchanged (η decreased from 98.8% to 98.6% when C_o increased from 50 to 200 mg/L). This could be due to the benefit of higher mass transfer efficiency which promoted the better absorption of ozone into the liquid.

3.3. Influence of Rotor Speed

In this investigation, an experiment was carried out with a rotating speed ranging from 300 to 1200 rpm because the HP2R is constructed with a maximum designed rotor rotational speed of 1375 rpm. Fig. 5 shows the effect of rotor speed on MB treatment efficiency.

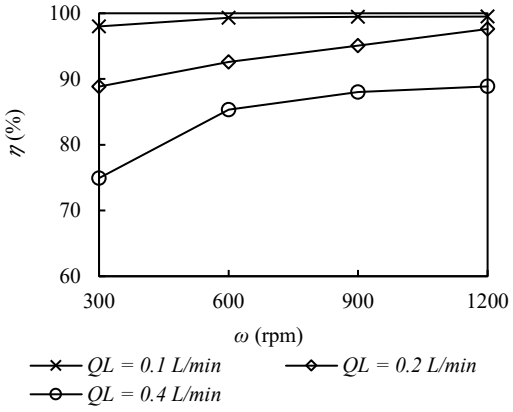

Fig. 5. Effect of rotor speed on decolorization efficiency

Fig. 5 shows that the decolorization had an increasing trend when the rotational speed increased from 300 to 1200 rpm at three different liquid flow rates. At Q_L = 0.1 L/min, the methylene blue treatment efficiency reached 98.0 to 99.5% when ω increased from 300 to 1200 rpm.

The increase in rotating speed improved the efficiency of methylene blue decolorization in wastewater due to enhanced centrifugal force which increased the turbulence of the liquid in HP2R, thus the liquid dispersed into thinner films and smaller droplets, consequently, increasing the area of gas-liquid mass transfer [17]. The greater ozone dissolved in wastewater led to a higher methylene blue efficiency. However, the retention time of the liquid in the packing decreased as the rotational speed increased ($\omega > 600$ rpm), which resulted in the ozone absorption rate and stable decolorization rate. This was similarly mentioned in the study of Liu *et al.* [19] on the inactivation of *E.coli* by ozone in RPB and Zequan *et al.* [20] about Ozonation of Phenol with O_3/Fe(II) in Acidic Environment in a rotating packed bed.

3.4. Influence of the Q_L

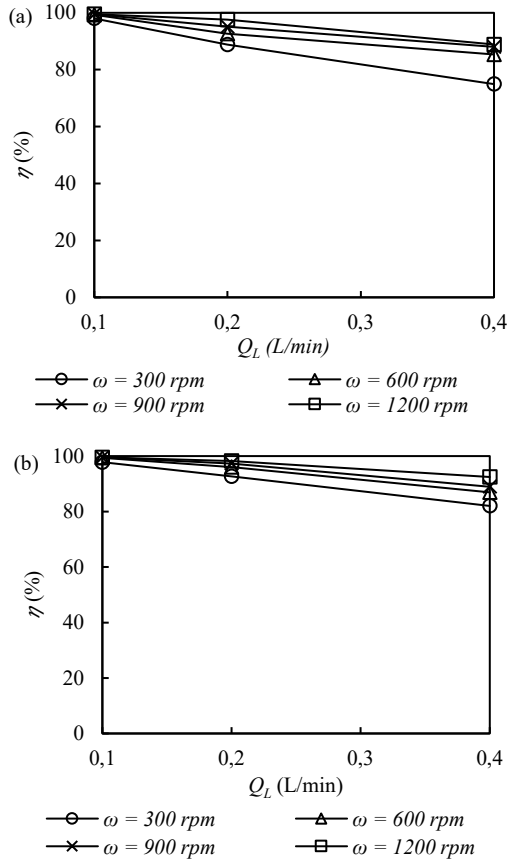

Fig. 6. Effect of Q_L on decolorization efficiency at (a) Q_G = 0.5 L/min and (b) Q_G = 1 L/min

The effect of the Q_L was presented in. Fig. 6. At Q_G = 0.5 L/min, when the Q_L increased from 0.1 to 0.4 L/min, the decolorization efficiency decreased from 98.0 to 74.9% at the rotational speed ω = 300 rpm. At ω = 1200 rpm, decolorization efficiency reduced from 99.5 to 88.9%. This trend was also noticed at Q_G = 1 L/min. It showed that the decolorization efficiency decreased with increasing Q_L which resulted in a shorter liquid residence time in the HP2R, which reduced the contact time between ozone

and methylene blue solution. Similar results were shown in the study of Chia *et al.* (2003) [18], Zequan *et al*. (2012) [20], and Xu *et al.* (2023) [17]. Besides, when increasing the gas flow from 0.5 to 1 L/min, the decolorization efficiency increases. The explanation for this is that a high input ozone concentration can produce a strong driving force for the mass transfer of ozone to water [20]. Due to ozone's limited solubility in water, increasing driving force can improve ozone absorption and the reaction between ozone and MB solution, boosting therapeutic effectiveness.

3.5. Decolorization Circulation Experiment

The circulation experiment was performed with the rotational speed ω = 600 rpm, the Q_L = 0.4 L/min, and the two Q_G were 0.5 L/min and 1 L/min. The results of the experiment on the methylene blue treatment efficiency showed in Fig. 7.

The results of the circulation experiment showed the superior methylene blue treatment efficiency of the combination ozonation and HP2R compared with conventional ozonation. In Q_G = 0.5 L/min, for the conventional ozonation, the decolorization efficiency reached 51.25% (after 5 minutes) and increased to 80.6% after 10 minutes.

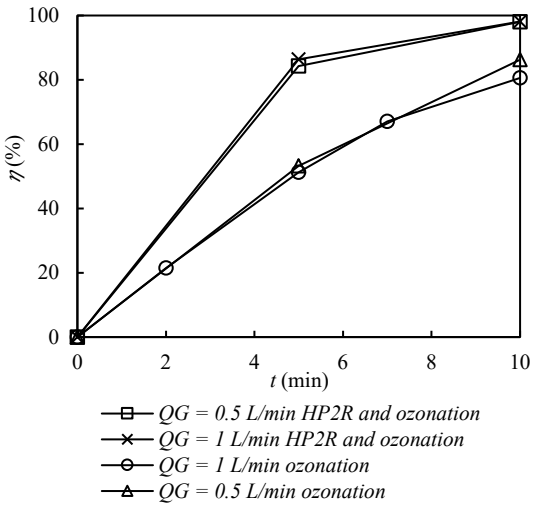

Fig. 7. Results of the decolorization circulation experiment

The decolorization efficiency of the combination HP2R and ozonation was 84.3% in the first cycle, in the second cycle the treatment efficiency is slightly better (η = 87.5%), the total time for two cycles was 10 minutes with an overall efficiency of 98.1%. The treatment efficiency was maintained when increasing the Q_G from 0.5 to 1 L/min. It was found that the second cycle methylene blue treatment efficiency tended to increase slightly compared to the first cycle, which was explained by the presence of excess ozone in the wastewater after 1st cycle and which improved the MB treatment efficiency of the 2nd cycle.

4. Conclusion

The removal of methylene blue from textile dyeing wastewater by ozonation in the HP2R was comprehensively investigated in this experiment. With the variation of parameters such as pH (2-10), initial MB (50-200 mg/L), ω (300-1200 rpm), and Q_L (0.1 to 0.4 L/min), the results the decolorization efficiency varied from 75.0 to 99.5%. The liquid flow rate and initial dye concentration had the most effect on the decolorization efficiency since their variation directly altered the MB loading rate into the HP2R system. By using the HP2R equipment, the decolorization efficiency after a single continuous cycle was equivalent or even higher than that obtained by the conventional ozonation process after 30 minutes. Multiple recirculation of wastewater into the ozonation using HP2R could even resulted in significantly low MB concentration in the output, where MB decreased from 112.0 mg/L to 2.1 mg/L after 2 cycles 10 min with total 2 L of wastewater. The results indicated that the advanced oxidation process using O_3 via HP2R benefits the MB treatment over the conventional process by minimize the hydraulic retention time, lessen the O_3 and electrical consumption. However, further effort should be focus on the investigation of O_3 absorption, O_3 tail gas, and technical-economical assessment of generator and rotor engine in the future study with HP2R.

Acknowledgments

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