Degradation of Reactive Blue 19 (RB19) by a Green Process Based on Peroxymonocarbonate Oxidation System

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1. Introduction

Reactive dyes play an important role in textile industries. However, they may cause serious environmental problems as they are often nonbiodegradable and toxic to aquatic systems due to their complex aromatic molecular structures [1]. Once being discharged to the environment without destructive treatment, these water-soluble dyes can remain for a long time and alter the quality of water bodies by preventing light penetration and hindering photosynthesis, thereby affecting the ecosystems [2]. Among reactive dyes, anthraquinone dyes contain reactive groups with reinforced structures, which makes them difficult to degrade naturally and can be bioaccumulative in animals [3]. For example, an estimated half-life of C.I. Reactive Blue 19 at pH 7.0 and 25°C based on kinetic studies is ca. 46 years [4]. The concern over the ecotoxicity of these reactive dyes has led to the need to develop more efficient methods for their remediation from industrial effluents before discharging to water bodies. Textile wastewater treatment is not only decolorizing but also degrading and mineralizing dye molecules.

Among technologies for dye treatment, advanced oxidation processes (AOPs) have attracted increasing attention due to their efficiency and ability to thoroughly remove pollutants from wastewater effluents. These AOPs base on the strong activity of in situ formed free radicals, among which the most common radical is •OH with a redox potential of +2.8 V. Thanks to their strong oxidizing capacity, organic substances can be completely mineralized into CO₂ and H₂O. Some typical processes for active radical
generation include Fenton, peroxone, H₂O₂/UV, O₃/UV, and TiO₂/UV.

A photochemical-based AOP of textile wastewater containing RB19 dye has been studied using the UV/K₃S₂O₈ system [5]. The results showed that only 50% of RB19 dye was removed after 5 hours in the dark system, while the UV-irradiated one gave a complete color removal in less than 30 min and 78.5% COD has been removed after 3 hours of irradiation time. The decolorization rate was fitted to the pseudo-first-order kinetic model regarding dye concentration. Another study on UV irradiation effect on RB19 decolorization conducted by Tehrani et al. who compared the RB19 decolorization efficiency by ozonation and UV-enhanced ozonation revealed that a UV irradiation by mercury lamp only increased COD removal efficiency, not the decolorization efficiency [6].

In advanced oxidation processes, besides *OH, *O₂−, and ¹O₂ radicals, other oxidizing agents such as HCO₃⁻, *CO₂, and HCO₂⁻ also play a crucial role in oxidation performance [7]. Peroxymonocarbonate (HCO₃⁻) ion is known to be an active oxidant in the oxidation of organic compounds [8, 9]. This oxidizing agent was proved to have 100–500 times stronger reactivity in sulfide organic oxidation compared to hydrogen peroxide [10]. Although *CO₂ and HCO₂⁻ radicals are reported to be less reactive than *OH, their occurrence at higher concentration and longer lifetime may provide sufficient oxidation potential. Another advantage of *CO₂⁻ and HCO₂⁻ radicals is a simple production technique.

One of the major benefits of the oxidation process based on the peroxymonocarbonate system is its greenerness toward the environment. As it can be prepared by mixing H₂O₂ with HCO₃⁻ or CO₂⁻, the oxidation process does not require neither expensive nor toxic chemicals nor energy demand. Furthermore, it does not generate secondary sludge waste or require chemicals to adjust pH as compared to the Fenton process.

The aim of this work was two-fold: (i) to evaluate the effectiveness of in situ generated HCO₃⁻ oxidizing agent in the HCO₃⁻—H₂O₂ system in degrading the RB 19 textile dye under different experimental conditions and (ii) to verify the products of RB19 degradation by oxidation system based on HCO₃⁻.

2. Materials and Experimental Procedure

2.1. Chemicals and Apparatus. Reactive Blue 19 dye was purchased from Sigma-Aldrich. Other chemicals and reagents used in this study were of analytical grade and used as received without any further purification.

The RB19 degradations were monitored by measuring RB19 absorbance at 588 nm using a Biochrom Libra S60 UV-Vis Spectrophotometer. The degradation efficiency was calculated using the following equation: RB19 degradation (%) = (C₀ − Cₜ)/C₀ × 100%, where C₀ and Cₜ are the initial and remaining RB19 concentrations (ppm) at time t (min), respectively. These concentrations were determined using the standard curve Abs = (8.4 ± 0.1) × 10^3 C_{RB19} (mg/L) (R² = 0.999) with the LOD and LOQ of 0.7 and 2.3 mg/L, respectively. pH values were measured by a Lab850 pH meter (with BlueLine 14 pH electrode). COD values were determined by the oxidation standard method [11]. Total carbon and total inorganic carbon were measured on a multi-N/C 2100 TOC analyser (Analytik Jena AG). A Shimadzu RP-HPLC equipped with a PDA-M20A detector was used to investigate the products of the degradation. A mixture of acetonitrile and phosphate buffer at pH of 4.7 (50/50, v/v) was used as a mobile phase with a flow rate of 1 mL/min, an injection volume of 20 μL, and an oven temperature of 40°C.

2.2. Experimental Procedure

2.2.1. In Situ Peroxymonocarbonate Preparation. Peroxymonocarbonate (HCO₃⁻) solutions were prepared in situ by mixing sodium bicarbonate with hydrogen peroxide at a molar ratio of 1:2 at ambient temperature. Kinetics of HCO₃⁻ formation was evaluated by monitoring HCO₃⁻ concentration at different times in three hours. In this experiment, HCO₃⁻ was analyzed by a modified iodometric titration method [12]. In which, HCO₃⁻ was first allowed to react with iodide at low temperature (below −10°C) to prevent the effect of H₂O₂. The formed I₂ was then titrated with thiosulfate using a starch indicator.

2.2.2. Degradation of RB19 by In Situ Generated HCO₃⁻ in the H₂O₂—HCO₃⁻ System. The effects of different parameters on the RB19 degradation were investigated as described in Table 1 including the metallic ion catalyst (trials 1–9), the pH (trials 10–13), the HCO₃⁻ concentration (trials 14–18), the Co²⁺ catalyst concentration (trials 19–22), and UV irradiation (trials 24–28). The RB19 concentration was 100.0 mg/L in all trials. Each trial was replicated three times. In trials 1–5, 8–22, and 26, the mixture of HCO₃⁻ and H₂O₂ with a molar ratio of 1 : 2 was prepared in 50 minutes before adding to the RB19 solution to conduct the dye decolorization. For trials 1–23, the reactions were conducted in a 250 mL batch reactor controlled at 26 ± 1°C using a thermostab and thoroughly mixed by a magnetic stirrer as depicted in Figure 1(a). For trials 24–28, the reaction solutions were irradiated with the UVC light (254 nm, 12 W) in the UV chamber regulated by a thermostab as described in Figure 1(b). The flow speed through the UV chamber was maintained at 100 mL/min using a circulating pump. Dye decolorization was monitored by sampling at certain intervals and measuring light absorbance at the wavelength of 592 nm.

3. Results and Discussion

3.1. Formation of Peroxymonocarbonate. The peroxymonocarbonate ion was produced in situ by the reaction of HCO₃⁻ and H₂O₂ according to the following reversible reaction [13]:

\[
\text{HCO}_3^- + \text{H}_2\text{O}_2 \leftrightarrow \text{HCO}_4^- + \text{H}_2\text{O} \tag{1}
\]

To determine the maximum formation of HCO₃⁻ formed in situ in the system, the variation of HCO₄⁻ concentrations
with time at a HCO$_3^-$: H$_2$O$_2$ molar ratio of 1:2 ([HCO$_3^-$] = 312.5 mM) has been studied and the result is given in Figure 2. It is revealed that HCO$_3^-$ was formed at the beginning, reached a maximum concentration at around 50 minutes, and then slowly decreased. This phenomenon can be explained by the decomposition of HCO$_3^-$ after formation.

Therefore, the dye decolorization experiments by the HCO$_3^-$ agent in this study were carried out by mixing H$_2$O$_2$ with HCO$_3^-$ 50 minutes before adding to the dye solution.

### 3.2. RB19 Degradation by H$_2$O$_2$– HCO$_3^-$ System

#### 3.2.1. Effect of Metallic Ion Catalysts

The effect of metallic ion catalysts (i.e., Ni$^{2+}$, Mn$^{2+}$, Zn$^{2+}$, and Co$^{2+}$) was investigated by performing trials 1–5 (Table 1). The result shown in Figure 3 reveals the most significant effect of Co$^{2+}$ on the degradation of RB19 by the H$_2$O$_2$– HCO$_3^-$ system. RB19 was degraded ca. 90% after 200 min in the presence of Co$^{2+}$, while the similar systems with other metallic ions such as Mn$^{2+}$, Zn$^{2+}$, and Ni$^{2+}$ and the bare system (without metallic ions) gave the degradation efficiency of less than 10%. This phenomenon can be explained by the great capability of Co$^{2+}$ in catalyzing oxidation reactions. In peroxycid solutions, Co$^{2+}$ enhances the decomposition of peroxycid to generate radicals [14, 15]. Applying the same mechanism, radicals are proposed to be formed by the following reactions:

\[
\text{HCO}_3^- + \text{CO}_2 \rightarrow \text{CO}_2^- + \text{Co}^{3+} + \text{OH}^- \quad (2)
\]

\[
\text{HCO}_3^- + \text{Co}^{2+} \rightarrow \text{CO}_3^- + \text{Co}^{3+} + \text{OH}^- \quad (3)
\]

Co$^{3+}$ is then reduced to regenerate Co$^{2+}$:

\[
\text{HCO}_3^- + \text{Co}^{3+} \rightarrow \text{CO}_3^- + \text{Co}^{2+} + \text{H}^+ \quad (4)
\]

These radicals then react with the organic compounds through many steps and eventually form CO$_2$ and H$_2$O. The oxidation process of organic compounds occurs continuously with a crucial role of the Co$^{2+}$/Co$^{3+}$ redox couple.

#### 3.2.2. Effect of pH

The effect of pH was investigated by varying pH values from 7 to 10 and keeping constant other parameters (trials 10–13). The result shown in Figure 5 revealed that the increase in pH value causes faster dye degradation. However, the

### Table 1: Conditions for RB19 decolorization by peroxymonocarbonate.

| Trial | pH | [HCO$_3^-$] (mM) | [H$_2$O$_2$] (mM) | [M$^{2+}$] catalyst (mg/L) | UV irradiation |
|-------|----|------------------|------------------|-------------------|---------------|
| 1     | 8  | 10               | 20               | —                 | No            |
| 2–5   | 8  | 10               | 20               | 0.1 (Ni$^{2+}$, Mn$^{2+}$, Zn$^{2+}$, Co$^{2+}$) | No |
| 6     | 8  | —                | 200              | 0.1 (Co$^{2+}$)   | No            |
| 7     | 8  | 100              | 200              | —                 | No            |
| 8     | 8  | 100              | 200              | 0.1 (Co$^{2+}$)   | No            |
| 9     | 8  | 100              | 200              | 0.1 (Co$^{2+}$)   | No            |
| 10–13 | 7, 8, 9, 10 | 10               | 20               | —                 | No            |
| 14–18 | 8  | 5, 10, 15, 25, 30| 40               | 0.1 (Co$^{2+}$)   | No            |
| 19–22 | 8  | 20               | 40               | 0.01, 0.02, 0.04, 0.06 (Co$^{2+}$) | No |
| 23    | 8  | —                | —                | —                 | No            |
| 24    | 8  | —                | —                | —                 | Yes           |
| 25    | 8  | —                | —                | —                 | Yes           |
| 26    | 8  | —                | —                | 0.1 (Co$^{2+}$)   | Yes           |
| 27    | 8  | 10               | 20               | —                 | Yes           |
| 28    | 8  | 10               | 20               | 0.1 (Co$^{2+}$)   | Yes           |
| 19–22 | 8  | 20               | 40               | 0.01, 0.02, 0.04, 0.06 (Co$^{2+}$) | No |
| 23    | 8  | —                | —                | —                 | No            |
| 24    | 8  | —                | —                | —                 | Yes           |
| 25    | 8  | —                | —                | —                 | Yes           |
| 26    | 8  | —                | —                | 0.1 (Co$^{2+}$)   | Yes           |
| 27    | 8  | 10               | 20               | —                 | Yes           |
| 28    | 8  | 10               | 20               | 0.1 (Co$^{2+}$)   | Yes           |
H2O2−HCO−3 system acts as a buffer solution of pH ca. 8. Therefore, the pH value used during this study was adjusted to 8 in order to be the closest to the intrinsic pH of the solution.

3.2.3. Effect of HCO−3 and Catalyst Concentrations: Kinetic Study of the RB19 Degradation Catalyzed by Co2+.

To study the effect of HCO−3 and Co2+ catalyst concentrations on the RB19 degradation, as well as to study reaction kinetics, the RB19 degradation was carried out either at different HCO−3 concentrations (trials 14–18) and at different Co2+ concentrations (trials 19–22). The RB19 concentration decreased as a function of time.

It can be seen from Figure 6(a) that RB19 degradation efficiency and rate remarkably increase with an increase in HCO−3 concentration from 5 mM to 30 mM while keeping constant other parameters (H2O2 and Co2+ concentrations, pH). It is because the increase in HCO−3 concentration leads...
to the increase in HCO$_3^-$ concentration according to the equilibrium reaction:

$$
\text{HCO}_3^- + \text{H}_2\text{O}_2 \rightarrow \text{HCO}_2^- + \text{H}_2\text{O} \quad (5)
$$

Similarly, Figure 7(a) shows a proportional relationship between the Co$^{2+}$ concentration and the RB19 degradation efficiency and reaction rate. The reason for this may be the increase in Co$^{2+}$ complexes as mentioned above.

The plots of $\ln(C_{0}/C_{t})$ vs. time shown in Figures 6(b) and 7(b) reveal that the kinetic degradation of RB19 is well fitted with the first-order kinetic model given by the following equation:

$$
\ln \left( \frac{C_0}{C_t} \right) = k_1 t. \quad (6)
$$

The reaction rate of RB19 degradation was assumed to be the pseudo-first-order kinetics with respect to RB19 as the following expression:

$$
v = -\frac{dC_t}{dt} = k_0 [\text{RB19}] [\text{HCO}_3^-]^{n_1} [\text{H}_2\text{O}_2]^{n_2} [\text{Co}^{2+}]^{n_3} [\text{H}^+]^{n_4} = k_1 [\text{RB19}], \quad (7)
$$

where

$$
k_1 = k_0 [\text{HCO}_3^-]^{n_1} [\text{H}_2\text{O}_2]^{n_2} [\text{Co}^{2+}]^{n_3} [\text{H}^+]^{n_4} \quad (8)
$$

is constant.

Integrating (7) gives (6): $\ln(C_{0}/C_{t}) = k_1 t$.

The pseudo-first-order rate constants, $k_1$ (min$^{-1}$), were calculated from the slope of the plots of $\ln(C_{0}/C_{t})$ vs. time $t$ and subsequently used to calculate the experimental order of HCO$_3^-$ and Co$^{2+}$.

1) Order of HCO$_3^-$. To determine the order of HCO$_3^-$, experiments were conducted at different HCO$_3^-$ concentrations when [H$_2$O$_2$], [Co$^{2+}$], and [H$^+$] were kept constant.

From equation (8),

$$
k_1 = k_2 [\text{HCO}_3^-]^{n_1}, \quad (9)
$$

where $k_2 = k_0 [\text{H}_2\text{O}_2]^{n_2} [\text{Co}^{2+}]^{n_3} [\text{H}^+]^{n_4}$.

Taking the logarithm of both sides of (9) gives

$$
\ln k_1 = n_1 \ln[\text{HCO}_3^-] + \ln k_2. \quad (10)
$$

2) Order of Co$^{2+}$. Similarly, the order of Co$^{2+}$ was determined when changing [Co$^{2+}$] and keeping concentrations of other species constant.

$$
\text{when}[\text{H}_2\text{O}_2], [\text{HCO}_3^-], \text{and}[\text{H}^+]\text{are constant}, \quad k_1 = k_3 [\text{Co}^{2+}]^{n_3}, \quad (11)
$$

where $k_3 = k_0 [\text{HCO}_3^-]^{n_1} [\text{H}_2\text{O}_2]^{n_2} [\text{H}^+]^{n_4}$.

Taking the logarithm of both sides of (11) gives

$$
\ln k_1 = n_3 \ln[\text{Co}^{2+}] + \ln k_3. \quad (12)
$$

The experimental orders of HCO$_3^-$, $n_1$, and Co$^{2+}$, $n_3$, were determined from the slope of the plot of $\ln(k_1)$ vs. $\ln[\text{HCO}_3^-]$. 

Figure 6: Dependence of (a) RB19 concentration and (b) $\ln(C_{0}/C_{t})$ vs. time at different HCO$_3^-$ concentrations and 40 mM H$_2$O$_2$, 0.1 mg/L Co$^{2+}$, and pH 8.
and ln($k_1$) vs. ln[Co$^{2+}$], respectively. The calculated data are shown in Table 2, and the result gives the experimental orders of HCO$_3^-$ and Co$^{2+}$ of 1.7 and 1.2.

### 3.2.4. Effect of UV Irradiation on the RB19 Degradation

The degradation of RB19 with the selected oxidation system (H$_2$O$_2$–HCO$_3^-$–Co$^{2+}$) was carried out without and with UV irradiation (trials 5 and 28) compared to other systems (trials 6–8 and 23–27). The results are shown in Table 3. With UV irradiation, the RB19 degradation efficiency significantly increased (ca. 90%) in all other systems compared to the ones without UV irradiation (less than 5%). This is due to the fact that UV radiation plays a crucial role in creating the radical *OH from H$_2$O$_2$ for the oxidation systems according to the photocatalytic mechanism as proposed in the literature [16]. Meanwhile, the highest RB19 degradation efficiency (97.6%) was obtained with the H$_2$O$_2$–HCO$_3^-$–Co$^{2+}$ system, slightly increased compared to the one without UV irradiation (79.9%). This strongly confirmed the degradation efficiency of the H$_2$O$_2$–HCO$_3^-$–Co$^{2+}$ system even without UV light.

### 3.3. Products of the RB19 Degradation

The COD values of the initial and final reaction solutions in which RB19 (0.1 g/L) was decomposed by the H$_2$O$_2$–HCO$_3^-$–Co$^{2+}$ system (after a reaction time of 60 min) were determined as 315 and 12.5 mg O$_2$/L, respectively. The TOC (total organic carbon) value was obtained from the subtraction of TC (total carbon) and TIC (total inorganic carbon) values measured for the final solution, giving the result of 15.3 mg/L which is in accordance with the COD value (Table 4).

These results suggest a good mineralization of RB19 by the H$_2$O$_2$–HCO$_3^-$–Co$^{2+}$ system with a COD removal efficiency of 96%.

Moreover, the RP-HPLC chromatograms of the RB19 degradation solutions using H$_2$O$_2$–HCO$_3^-$–Co$^{2+}$ system at reaction times of 10, 30, and 60 min were recorded at 280 nm (Figure 8(b)). It can be seen clearly that the intermediate products of RB19 degradation at a reaction time of 10 min and 30 min are less polar and eluted before the remained RB19 (zoom in the lower inset in Figure 8(b) compared with the chromatogram of pure 1 mg/L RB19 solution shown in Figure 8(a) with a retention time of 8.2 minutes). The unretained components are eluted at the time corresponding to the reaction time.
to the travel time of the mobile phase of 1.5 min. Most of these products are not observed after the reaction time of 30 min, and the chromatogram obtained for the reaction solution at 60 min is flattened (the upper inset). This result strongly supports the effectiveness of the RB19 degradation to a complete mineralization by the $\text{H}_2\text{O}_2 - \text{HCO}_3^- - \text{Co}^{2+}$ system.

### Table 3: The effect of UV irradiation on the RB19 degradation.

| Oxidation system          | RB19 degradation efficiency at 30 min reaction time (%) |
|---------------------------|----------------------------------------------------------|
|                           | Non-UV irradiation | UV irradiation |
| RB19                      | 0.0               | 3.7 ± 0.3     |
| RB19-$\text{H}_2\text{O}_2$ | 0.0               | 83.6 ± 3.6    |
| RB19-$\text{H}_2\text{O}_2$-$\text{Co}^{2+}$ | 1.8 ± 0.2         | 91.1 ± 2.9    |
| RB19-$\text{H}_2\text{O}_2$-$\text{HCO}_3^-$ | 4.7 ± 2.7         | 96.7 ± 2.3    |
| RB19-$\text{H}_2\text{O}_2$-$\text{HCO}_3^-$-$\text{Co}^{2+}$ | 79.9 ± 3.5     | 97.6 ± 3.1    |

### Table 4: Determination of COD and TOC of RB19 degradation.

|                          | COD (mg $\text{O}_2$/L) | TC (mg/L) | TIC (mg/L) | TOC (mg/L) |
|--------------------------|--------------------------|-----------|------------|------------|
| RB19-$\text{H}_2\text{O}_2$-$\text{HCO}_3^-$-$\text{Co}^{2+}$ initial | 315 ± 0.5                | —         | —          | —          |
| RB19-$\text{H}_2\text{O}_2$-$\text{HCO}_3^-$-$\text{Co}^{2+}$ final      | 12.5 ± 0.3               | 141.5 ± 0.9 | 126.2 ± 0.9 | 15.3 ± 0.2 |

### Figure 8: (a) The chromatogram of pure RB19 solution (1 mg/L); (b) the chromatograms of the RB19 degradation solution by the $\text{H}_2\text{O}_2 - \text{HCO}_3^- - \text{Co}^{2+}$ system at different reaction times ($t = 10$, 30, and 60 min); the upper inset: the chromatogram recorded at a reaction time of 60 min; the lower inset: zoom-in view of the chromatogram recorded at a reaction time of 10 min.

4. Conclusions

In the present work, the degradation efficiency of RB19 reactive dye has been proved to be strongly affected by the $\text{HCO}_3^-$ concentration and the presence of metallic ion catalysts, especially $\text{Co}^{2+}$. This improvement can be explained by an enhancement in radicals generated by the $\text{Co}^{2+}/\text{Co}^{3+}$
redox couple. The in situ formation of peroxymonocarbonate ion $\text{HCO}_3^-$ is responsible for the oxidation efficiency of the system. This $\text{HCO}_3^-$ ion was determined to reach a maximum concentration after a $\text{H}_2\text{O}_2$–$\text{HCO}_3^-$ mixing time of 40 min and remain stable for ca. 10 min. This kind of information is essential for practical applications of the system. The degradation kinetics of RB19 was determined to be the first order in RB19 dye while reaction orders of $\text{HCO}_3^-$ and $\text{Co}^{2+}$ catalyst were 1.7 and 1.2, respectively. The products of RB19 degradation were investigated by RPHPLC analyses and COD and TOC measurements, which suggest a complete mineralization of RB19 by the $\text{H}_2\text{O}_2$–$\text{HCO}_3^-$–$\text{Co}^{2+}$ system.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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