Original Article

Mechanistic Insights into Crystal Violet Degradation by Peroxymonocarbonate-Based Oxidation Systems

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Abstract: In this study, peroxymonocarbonate (PMC), formed directly from the reaction between H_2O_2 and HCO_3^-, was applied to decolorize Crystal Violet (CV), a widely used dye in the textile industry. Experimental results showed that PMC can decolorize CV almost completely in 30 minutes, superior to the H_2O_2 or H_2O_2/Co^{2+} systems. The effects of reactant concentration, catalyst, pH, co-existing anions, and UV irradiation were systematically investigated and optimized. The results showed that at concentrations of 8 mM HCO_3^-, 20 mM H_2O_2, 1.7 μM Co^{2+}, and pH 8.5 (inherent pH), under UV irradiation, the PMC system can completely mineralize organic substances and is not affected by anions. Experiments using ROS scavengers showed that the oxidizing agents generated in the PMC system play an important role in CV decolorization, in which the superoxide radical ($O_2^{\bullet-}$) contributes more to the CV decolorization process compared to the carbonate radical ($CO_3^{\bullet-}$), while the hydroxyl radical ($^{\bullet}OH$) and singlet oxygen ($^1O_2$) are almost negligible.

Keywords: Crystal Violet, peroxymonocarbonate, AOPs, reactive oxygen species.

1. Introduction

Crystal Violet (CV), a triphenylmethane cationic dye, is extensively utilized in global industries such as textiles, printing, aquaculture, and biological staining due to its vibrant purple hue, high color fastness, and economic viability [1-3]. However, approximately 10–15% of CV remains unbound to fabric fibers during the dyeing process and is thus released into wastewater streams [4, 5]. The inherent stability of CV's conjugated aromatic structure makes it highly recalcitrant to natural degradation processes, leading to its persistence in aquatic environments [3, 6]. The accumulation of CV in water bodies elevates chemical and biological oxygen demand (COD, BOD), depletes dissolved oxygen (DO) levels, and inhibits photosynthesis, posing significant ecological threats [5, 7]. Beyond its environmental impact, CV is a recognized health hazard, exhibiting cytotoxic, mutagenic, and potentially carcinogenic properties in humans and other organisms, which necessitates the development

of effective and sustainable remediation technologies [2, 4].

To date, various strategies have been employed to treat dye-contaminated wastewater, each with inherent limitations. Biological methods employing bacteria, fungi, or algae, while environmentally friendly and cost-effective, often exhibit low removal efficiency, particularly at high dye concentrations or under fluctuating pH conditions [2, 8, 9]. In contrast, advanced oxidation processes (AOPs), including Fenton/photo-Fenton, ozonation, persulfate activation, and photocatalysis, have proven effective for CV degradation by generating powerful, non-selective hydroxyl radicals ($^{\bullet}OH$). Despite their efficacy, the widespread application of these AOPs is often constrained by significant drawbacks such as high operational and chemical costs, the generation of secondary iron sludge (in Fenton-based systems), and substantial energy consumption, particularly for UV-based methods [1, 6, 7, 10-14]. These challenges highlight the ongoing need for more sustainable, efficient, and cost-effective alternatives.

In this context, peroxymonocarbonate (PMC, HCO_4^-), formed from the reaction of hydrogen peroxide with bicarbonate or dissolved carbon dioxide, has emerged as a promising and environmentally benign oxidant [15-18]. PMC can be efficiently activated by various means, including UV irradiation or transition metal ion catalysts (e.g., Co^{2+}, Fe^{2+}), to produce a diverse spectrum of reactive oxygen species (ROS). This includes not only $^{\bullet}OH$ but also carbonate radicals ($CO_3^{\bullet-}$), superoxide radicals ($O_2^{\bullet-}$), and singlet oxygen ($^1O_2$), among others [19-22]. The synergistic action of this multi-ROS system offers a powerful pathway for the rapid and potentially complete mineralization of persistent organic pollutants. Despite this considerable potential, systematic investigations into the application of PMC-based systems for CV degradation remain notably scarce in the literature. Key aspects, such as the optimization of operational parameters, the identification of degradation intermediates, and the mechanistic contributions of the different ROS, have not been comprehensively elucidated.

This study, therefore, aims to bridge these critical knowledge gaps by conducting a thorough investigation of CV degradation within activated PMC systems. We systematically evaluate the influence of essential parameters, including catalyst type, initial solution pH, concentration of H_2O_2 and HCO_3^-, and the effects of co-existing anions and UV irradiation.

The degradation pathway and efficiency are monitored through UV–Vis spectroscopy and COD analysis, while the roles of specific ROS are mechanistically probed using selective chemical scavengers. The findings provide crucial insights into optimizing the efficiency of PMC-based AOPs and demonstrate their potential for treating wastewater laden with other recalcitrant cationic dyes.

2. Materials and Methods

2.1. Materials

Sodium bicarbonate ($NaHCO_3$), hydrogen peroxide (H_2O_2, 30%), cobalt(II) nitrate, copper(II) nitrate, manganese(II) nitrate, nickel(II) nitrate, zinc(II) nitrate, iron(II) nitrate, sodium nitrate, sodium sulfate, sodium chloride, dimethylamine (DMA), terephthalic acid (TA), sodium azide (NaN_3), p-benzoquinone (BQ), ascorbic acid (AA), Crystal Violet were purchased from Sigma-Aldrich. All chemicals were of analytical grade and used without further purification.

2.2. Methods

2.2.1. Investigation of Optimal Conditions for CV Decolorization by the PMC System

The influence of various operational parameters on CV degradation efficiency was systematically investigated by varying one variable at a time while keeping others constant.

The investigated parameters included: i) transition metal ions (M^{2+}) (trials 1÷6); ii) composition of the oxidation system (trials 1, 6, 7, 8); iii) initial concentrations of H_2O_2 and $NaHCO_3$ (trials 9÷11); iv) initial solution pH (trials 6, 12÷15); v) co-existing anions (trials 16÷18); and vi) UV irradiation using a 254-nm UVC lamp (12 W) (trials 19÷22). The specific experimental conditions for each set of experiments are detailed in Table 1.

All dye degradation experiments were conducted in a 250-mL glass reactor thermostated at 25 ± 1 °C with continuous magnetic stirring. In a typical run, the reaction was initiated by adding the required amounts of H_2O_2, $NaHCO_3$, and M^{2+} catalyst to a 20-ppm (0.049 mM) CV solution. At predetermined time intervals, 3-mL aliquots were withdrawn to analyze the residual CV concentration by measuring the absorbance at its λ_{max} of 590 nm using a Biochrom Libra S60 UV-Vis spectrophotometer. To ensure data reliability, all experiments were conducted in triplicate.

Table 1. Experimental conditions for CV decolorization by the PMC system

Trials	pH	HCO_3^- (mM)	H_2O_2 (mM)	M^{2+} catalyst	Anion added (0.1 M)	UV (**)
1÷6	8.5	8	20	No catalyst; Ni^{2+}; Mn^{2+}; Fe^{2+}; Cu^{2+}; Co^{2+} (0.1 ppm)	No	No
7	8.5	0	20	0	No	No
8	8.5	0	20	Co^{2+} (1.7 μM)	No	No
9÷11	8.5	4; 6; 10	10; 15; 25	Co^{2+} (1.7 μM)	No	No
12÷15	6.5; 7; 9; 10(*)	8	20	Co^{2+} (1.7 μM)	No	No
16÷18	8.5	8	20	Co^{2+} (1.7 μM)	Cl^-; SO_4^{2-}; NO_3^-	No
19	8.5	0	10	0	No	UV
20	8.5	0	10	Co^{2+} (1.7 μM)	No	UV
21	8.5	4	10	0	No	UV
22	8.5	4	10	Co^{2+} (1.7 μM)	No	UV

(*) adjusted using 0.1 M NaOH or 0.1 M HCl solutions.
(**) Experiments with UV irradiation were performed as described in our previous work [25].

The CV concentration was calculated from the calibration curve equation: $A_{590nm} = (0.0563 \pm 0.0005) \times C_{CV}$ (μM) with $R^2 = 0.9998$ in a linear range of 4.90 – 53.92 μM (2 – 22 ppm). The limit of detection (LOD) and limit of quantitation (LOQ) were 1.41 μM and 4.70 μM, respectively.

The CV degradation efficiency was calculated using the following equation:

$$H\% = \frac{C_0 - C_t}{C_0} \times 100$$

where C_0 and C_t are the CV concentrations at the initial time and time t, respectively.

2.1.2. Mineralization and Spectral Analysis

The extent of CV mineralization by the PMC system was evaluated by measuring the Chemical Oxygen Demand (COD) of the solution before and after treatment. COD was determined according to the standard method ISO 15705:2002.

To monitor changes in the dye's molecular structure during degradation, full-range UV-Vis absorption spectra of the samples were recorded from 190 to 700 nm.

2.1.3. ROS Scavenging Experiments

To identify the primary ROSs responsible for CV degradation, scavenging experiments

were performed under previously optimized conditions as described in 2.2.1 (NaHCO₃, H₂O₂, Co^{2+} concentrations) in the presence of specific ROS scavengers. The following scavengers were used at their pre-optimized concentrations: 5 mM AA as a general ROS scavenger, 0.371 mM TA for $^{\cdot}OH$ radicals, 2.65 mM DMA for $CO_3^{\cdot-}$ radicals, 10 mM NaN_3 for $^1O_2$, and 1 mM BQ for $O_2^{\cdot-}$ radicals. The degradation efficiency of CV was compared in the absence and presence of each scavenger.

3. Results

3.1. Influence of Various Factors on the CV Degradation

a. Effect of metal ion catalysts

The potential catalytic effect of various transition metal ions (Ni^{2+}, Cu^{2+}, Fe^{2+}, Mn^{2+}, and Co^{2+}) on CV degradation was evaluated. Surprisingly, the catalyst-free PMC system was itself highly effective, achieving nearly 100% decolorization of CV within 25 minutes (Figure 1a). The addition of the metal ions offered no discernible enhancement (even with Co^{2+}), as the degradation kinetics were nearly identical across all systems, including the control. This suggests that the intrinsic oxidative power of the PMC system is sufficient for rapid CV degradation under these specific conditions, effectively masking any potential catalytic contributions.

It is well known that the central carbon atom of CV is easily attacked by anions such as CN^-, OH^-, that led to the formation of Leuco Crystal Violet (LCV). This product is colourless due to breaking of high conjugated π system [23, 24]. In PMC system, the primary degradation/decolorization pathway can be directly driven by peroxymonocarbonate (HCO_4^-), which is non-dependent on the presence of metal ions. High concentration of HCO_4^- contributes to most of decolorization compared to free radicals. Then unclear catalytic activity was observed for tested cations in this study.
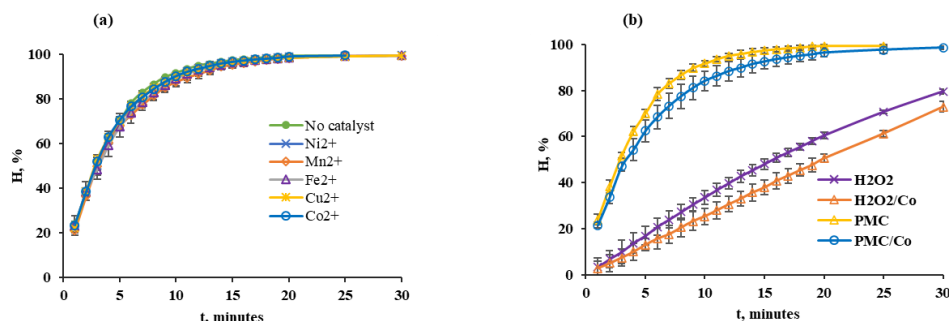

Figure 2. (a) Catalytic activity of various metal ions (Ni^{2+}, Cu^{2+}, Fe^{2+}, Mn^{2+}, and Co^{2+}) in CV degradation; (b) Comparison of Co^{2+} performance in the H_2O_2 system versus PMC system.

Notably, this finding contrasts sharply with previous reports on other dyes, such as Reactive Blue 19 (RB19) [25] or Rhodamine B (RhB) [26], where a Co^{2+} catalyst was essential for high efficiency in PMC systems.

To highlight the efficacy of the PMC system, its performance was compared to an H_2O_2 system, with and without Co^{2+} catalyst (Figure 3b). The H_2O_2 system alone, even with Co^{2+} acting as a Fenton-like catalyst, only achieved ~70% decolorization after 25 minutes. In contrast, the PMC system achieved complete decolorization in the same timeframe. This result clearly demonstrates the significantly enhanced oxidative capacity of the PMC system over conventional H_2O_2-based processes for CV degradation.

b. Effect of H_2O_2 and NaHCO₃ concentrations

The effect of reactant concentration on CV degradation was investigated by varying the initial amounts of HCO_3^- and H_2O_2 while maintaining a constant molar ratio (HCO_3^-: H_2O_2 = 1: 2.5). As shown in Figure 4a, increasing the initial HCO_3^- concentration from 4 mM to 10 mM significantly accelerated the rate and improved the final efficiency of CV decolorization. This trend is attributed to a higher rate of HCO_4^- formation at greater reactant concentrations, which is consistent with previous studies on RB19 and RhB degradation by the PMC systems [25, 26]. For instance, at a HCO_3^- concentration of 10 mM, over 99% degradation was achieved in just 20

minutes, whereas at 4 mM, the efficiency only reached 87% after 30 minutes. However, the kinetic data also revealed a point of diminishing returns. As seen in Figure 4b, the improvement in degradation efficiency became marginal when the HCO_3^- concentration was increased from 8 mM to 10 mM, with both systems showing nearly identical performance after 10 minutes. This indicates that the system becomes saturated with oxidants at concentrations above 8 mM. Therefore, to ensure high efficiency while minimizing chemical consumption, an initial HCO_3^- concentration of 8 mM (corresponding to 20 mM of H_2O_2) was selected as the optimal condition for subsequent experiments.
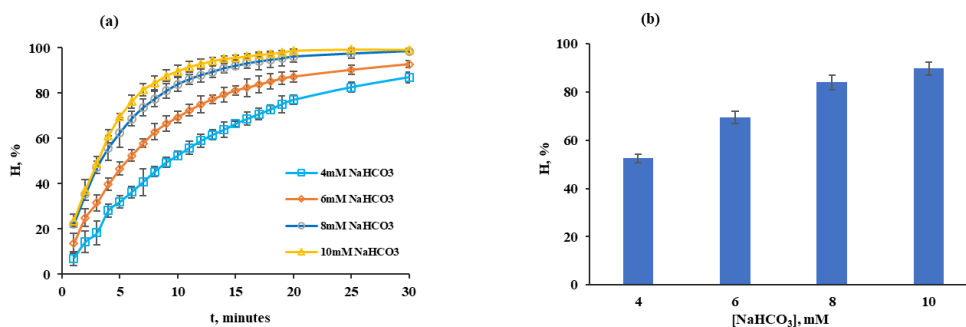

Figure 4. Effect of initial NaHCO$_3$ concentration on CV degradation at a constant HCO_3^-:H_2O_2 molar ratio of 1:2.5. (a) Degradation efficiency over time and (b) final efficiency as a function of NaHCO$_3$ concentration.

c. Effect of Initial pH

The PMC system inherently creates a weakly alkaline environment (pH 8-9), which aligns well with the typically alkaline nature of textile wastewater. Consequently, the initial pH is a critical parameter governing the system's performance. In this study, we investigated the influence of pH at values of 6.5, 7.0, 8.5 (original pH of the system), 9.0, and 10.0. The results presented in Figure 5, demonstrate that the initial pH profoundly impacts both the rate and efficiency of CV decolorization.

At a slightly acidic pH of 6.5, the reaction was slow, achieving only 43% decolorization in 30 minutes. At neutral pH (7.0), the efficiency improved to 68% over the same period. A significant enhancement was observed in alkaline conditions. At the system's original pH

of 8.5, over 90% of the dye was decolorized within just 15 minutes. Optimal performance occurred at pH 9.0 and 10.0, where the reaction was extremely rapid, reaching 100% decolorization in just 3 to 7 minutes.
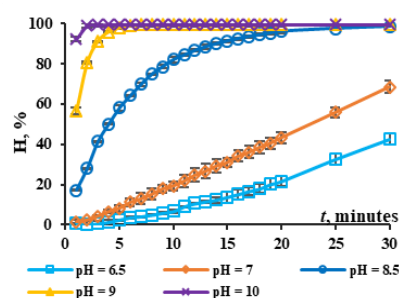

Figure 5. Influence of initial pH on the CV degradation efficiency by PMC-AOPs: pH 6.5, 7, 8.5, 9, 10.

These findings confirm that the PMC/Co^{2+} system is most effective in an alkaline environment. This pH dependency is attributed to the formation equilibrium of the key oxidant, HCO_4^-, from HCO_3^- and H_2O_2. This equilibrium is favored under alkaline conditions [20], leading to a higher concentration of HCO_4^- and subsequently a greater generation of potent ROSs that degrade the dye. This behavior is consistent with previous findings on the degradation of other dyes, such as RB19 and RhB [25, 26].

While the fastest rates were observed at pH 9-10, it is noteworthy that complete decolorization was still achieved within 30 minutes at the system's natural pH of 8.5. This condition offers a significant practical advantage by eliminating the need for chemical additives for pH adjustment. Therefore, to simplify the process and reduce chemical costs, the original pH of 8.5 was selected as the optimal condition for all subsequent experiments.

d. Effect of Co-existing Anions

Textile industry wastewater typically contains high concentrations of inorganic anions like sulfate (SO_4^{2-}), nitrate (NO_3^-), and chloride (Cl^-), which can act as ROS scavengers and inhibit the efficiency of AOPs. To evaluate the robustness of the PMC system, the effect of these three anions was investigated at a concentration of 0.1 M. As shown in Figure 6, the presence of SO_4^{2-}, NO_3^-, or Cl^- had a negligible effect on the decolorization of CV. The kinetic curves for the samples containing anions were nearly identical to the control sample (without added anions), all achieving over 95% color removal within 30 minutes.

This remarkable resistance to inhibition suggests that the primary reactive species in the PMC/Co^{2+} system are not easily scavenged by these common anions. It's well-established that anions like chloride are potent scavengers of $^•OH$ radicals. The lack of inhibition here implies that other species, play the dominant

role in the degradation process [27, 28]. These reactants are more selective and less susceptible to scavenging by anions compared to $^•OH$. This stability in a complex water matrix is a significant advantage of the PMC system, highlighting its strong potential for treating real industrial wastewater.
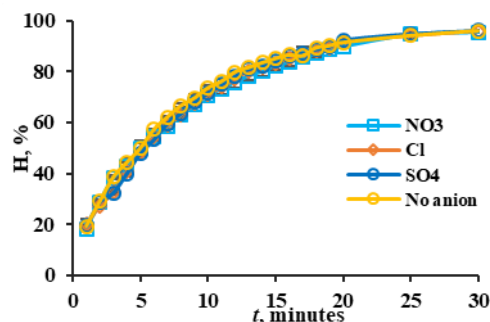

Figure 6. Influence of co-existing anions on the CV degradation efficiency by PMC-AOPs: SO_4^{2-}, NO_3^-, and Cl^- at 0.1 M concentration.

e. Effect of UV Irradiation

To explore potential synergistic effects, the performance of the optimized PMC/Co^{2+} system was evaluated under UV irradiation and compared with other related systems. The results in Figure 7 reveal that UV irradiation significantly accelerates decolorization in systems without the Co^{2+} catalyst. For instance, in the H_2O_2-only system, UV light induced a 3-fold increase in degradation efficiency after 10 minutes. This enhancement is due to the photolysis of hydrogen peroxide ($H_2O_2 + h\nu \rightarrow 2^•OH$), which generates highly reactive hydroxyl radicals. A similar, though less pronounced, enhancement was seen in the H_2O_2/HCO_3^- system, indicating that UV also promotes the ROS generation from HCO_4^-.

However, a key finding is that for the Co^{2+}-containing systems (H_2O_2/Co^{2+} and $H_2O_2/HCO_3^-/Co^{2+}$), UV irradiation provided unclear additional benefit. The decolorization rates were nearly identical with or without UV light. This suggests that the catalytic activation of peroxides by Co^{2+} is so rapid and efficient that it is the dominant degradation pathway.

The reaction rate is likely limited by the speed of the catalytic cycle itself, rendering the contribution from UV-induced radical formation redundant. This result further reinforces the conclusion from section 3.1.a.
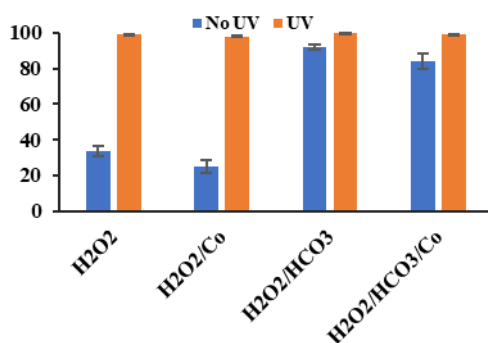

Figure 7. Influence of a 10-minute UV irradiation on the CV degradation efficiency by H_2O_2 and H_2O_2/HCO_3^- systems with and without Co^{2+}.

3.2. Evaluation of CV Mineralization and Degradation

To determine whether the CV dye was merely decolorized or fully broken down, the extent of mineralization was evaluated using COD analysis and UV-Vis spectroscopy. First, the COD of a 20-ppm CV solution was measured before and after a 120-minute treatment with the optimized $H_2O_2/HCO_3^-/Co^{2+}$ system. The initial COD was 47.2 mg O_2/L, which decreased dramatically to 2.6 mg O_2/L after the reaction. This corresponds to a COD removal efficiency of 94.5%, providing strong evidence that the complex organic dye was not just transformed but was extensively mineralized into simpler, less harmful substances.

Further insight into the structural breakdown of the dye molecule was provided by UV-Vis spectral analysis (Figure 8). The initial spectrum of the CV solution showed two key absorption peaks: one in the visible region at 590 nm, which produces the dye's characteristic purple color, and another in the UV region at ~260 nm, corresponding to the

stable aromatic rings in its structure. After 120 minutes of treatment, both the visible and UV peaks were completely eliminated. The disappearance of the 590 nm peak confirms the destruction of the dye's chromophore, which explains the rapid color removal. More significantly, the elimination of the 260 nm peak indicates the cleavage of the aromatic rings-a crucial and often difficult step in the degradation of organic pollutants.

In conclusion, the combination of spectroscopic data showing the destruction of the parent molecule and the near-total reduction in COD provides compelling evidence that the PMC/Co^{2+} oxidation system achieves near-complete mineralization of Crystal Violet.
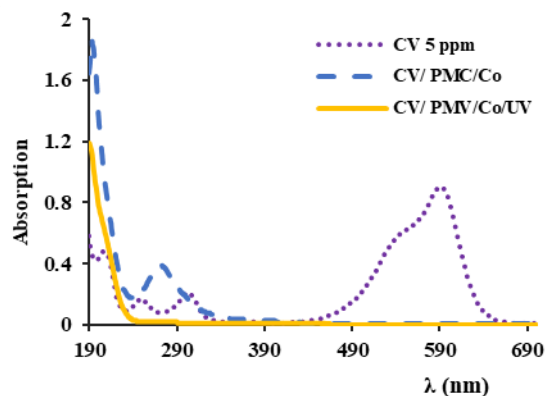

Figure 8. UV-Vis spectra of different solutions: a 5-ppm CV solution, CV solution treated by PMC/Co^{2+} system, and CV solution treated by $PMC/Co^{2+}/UV$ system.

3.3. Identification of Dominant ROSs

To elucidate the key ROS responsible for degrading Crystal Violet (CV), quenching experiments were conducted, with the results presented in Figure 9. First, the overall role of ROSs was confirmed. The control sample (no scavenger) showed a nearly complete decolorization, demonstrating the PMC system's high oxidative capacity. In contrast, the addition of AA, a broad-spectrum antioxidant, almost entirely halted the reaction,

indicating that the degradation process is overwhelmingly driven by ROS. To distinguish the contributions of specific species, selective scavengers were applied as described in section 2.1.3. The results revealed a clear hierarchy among the scavenged species. BQ exerted the most pronounced effect, reducing the decolorization efficiency by ~17%. DMA caused a moderate reduction of ~7%, whereas both TA and NaN$_3$ produced negligible inhibition (< 2%). These observations suggest that the relative importance of the ROS in CV degradation follows the order: $O_2^{\bullet-} > CO_3^{\bullet-} > {}^1O_2$, $^{\bullet}OH$.

Importantly, even the most effective selective scavengers only partially suppressed the reaction, implying that the majority of CV degradation is mediated by another highly potent oxidant not targeted by these scavengers. This dominant species is most likely HCO_4^-, a strongly oxidative species formed in the PMC systems. Notably, this trend contrasts with earlier reports, where carbonate radicals were found to play a major role in the degradation of other dyes, such as Reactive Blue 21 [27], Acid Orange 7 [28], Acid Orange 8 [29].
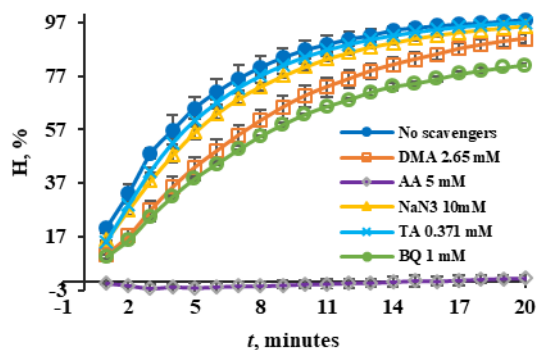

Figure 9. Identification of ROS contribution to CV degradation by PMC-based AOPs.

4. Conclusion

This study demonstrated that the PMC system is a highly effective advanced oxidation process for degrading and mineralizing Crystal Violet dye. The process operates optimally at its natural alkaline pH (~8.5) and remains efficient in the presence of common inorganic anions (SO_4^{2-}, NO_3^-, Cl^-), both with or without Co^{2+}. Additional energy inputs such as UV irradiation do not enhance decolorization but primarily promotes mineralization. Radical scavenging tests indicated that while $O_2^{\bullet-}$ and $CO_3^{\bullet-}$ play supporting roles, HCO_4^- is likely the dominant oxidant. Overall, the PMC system combines high efficiency, robustness, and operational simplicity, underscoring its strong potential for practical textile wastewater treatment.

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