
Employing in-situ hydrogen peroxide generation to alleviate aquatic impact from methylene blue-contaminated water

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Abstract The accumulation of dyes in aquatic environments poses significant ecological risks, and methylene blue (MB) is one such dye that requires effective removal. This study evaluated the efficiency of an electrolytic system for generating hydrogen peroxide (H₂O₂) to decolorize MB. The process was optimized by examining the effects of pH, applied current, and activated carbon (AC) weight using Response Surface Methodology (RSM). Results indicated a high coefficient of determination ($R^2 = 0.9446$), demonstrating the robustness of the predictive model for decolorization. Increasing applied current, oxidation time, and AC weight improved color removal, while lower pH levels enhanced the process, consistent with electro-Fenton principles. The maximum color removal achieved was 57.51% under optimal conditions. Additionally, three hydrogen peroxide production methods—bubbling, electrolysis, and electrolysis with Fe²⁺ addition—were compared. The results showed that while bubbling alone was less effective for color removal, both electrolysis and electrolysis with Fe²⁺ significantly increased color removal, with electrolysis and Fe²⁺ addition proving to be the most effective method. This study highlights the potential of an electrolytic system for efficient dye decolorization, offering a promising solution for treating dye-contaminated water.

Keywords: Activated carbon, Hydrogen peroxide, Response surface methodology

Introduction

The synthetic dye manufacturing industry is experiencing significant growth, driven primarily by high demand from the textile and textile sectors. This increase in production results in the annual generation of thousands of tons of synthetic dyes worldwide (Oladoye *et al.*, 2022). The release of these dyes into aquatic environments has emerged as a significant environmental concern, largely due to their potential to contaminate water sources (Quimada *et al.*, 2023). In particular, the dyeing and finishing processes in textile manufacturing are recognized as major contributors to aquatic pollution (Mehra *et al.*, 2021). Amid growing concerns over resource depletion and environmental degradation, such

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as the looming global energy crisis, industries are compelled to explore more sustainable practices and cleaner technologies (Arazo *et al.*, 2016). The enhanced need to manage environmental issues has made sustainability a focal area in the various fields (Javed *et al.*, 2024); this is true for management, as industries strive to implement more sustainable practices to mitigate environmental impacts.

Active methods for eliminating dyes and chemical oxygen demand (COD) from wastewater include techniques such as chemical coagulation, electrocoagulation, oxidation, photocatalytic degradation, and adsorption (Quimada *et al.*, 2024; Quimada *et al.*, 2020). Hydrogen peroxide (H_2O_2), an environmentally friendly chemical with both oxidizing and reducing properties, is utilized in various applications including organic compound synthesis, bleaching, wastewater treatment, and the degradation of hazardous organic wastes. Furthermore, H_2O_2 serves to supplement oxygen sources for increasing the bioremediation of contaminated aquifers (Bañuelos *et al.*, 2014).

Dyeing wastewater, a byproduct of multiple industrial processes such as dye production, textile manufacturing, leather processing, paper and plastic production, food processing, cosmetics, and rubber processing, is characterized by a high concentration of complex organic pollutants. This wastewater typically exhibits fluctuating water quality, a high COD level, deep and persistent color, and significant toxicity, posing a severe threat to both aquatic ecosystems and human health (Colades *et al.*, 2015).

Previous studies have explored various methods for generating H_2O_2 to address this issue, as H_2O_2 reacts effectively with organic pollutants. These methods include advanced oxidation processes, oxidation with activated carbon (AC) as a catalyst, pulsed discharge plasma systems, electrochemical oxidation, Fenton and electro-Fenton processes, and photoelectro-Fenton processes (Fayazi *et al.*, 2016; Georgi and Kopinke, 2005; Hidayu and Muda, 2016; Khataee *et al.*, 2011; Nurbazilah *et al.*, 2016; Quimada *et al.*, 2024).

One of the most effective in-situ methods for H_2O_2 production involves an enzyme-like process where homogeneous manganese (Mn) complexes catalyze the reaction between hydroxylamine and oxygen. However, manganese species can also significantly decompose H_2O_2 , reducing its effectiveness. To overcome this limitation, the use of supported metal catalysts such as gold (Au) and platinum (Pt) nanoparticles has been explored. While these methods achieve high H_2O_2 yields, the high cost and limited availability of precious metals make them less economically viable. In contrast, activated carbon, a widely used heterogeneous catalyst, offers a cost-effective and efficient alternative for in-situ H_2O_2 production. Due to its large specific surface area, porous structure, and ability to adsorb a wide range of organic pollutants, activated carbon not only facilitates the decomposition of H_2O_2 but also promotes hydroxyl radical

formation in the ozone oxidation system. This enhances the degradation of organic contaminants and improves wastewater treatment efficiency (Nurbazilah *et al.*, 2016). Additionally, activated carbon is economically feasible and easy to recycle.

Despite these advantages, the high consumption of H_2O_2 sets economic challenges, highlighting the need for cost-effective and technically feasible substitutes for applications like textile wastewater treatment. Traditionally, H_2O_2 is produced industrially through the anthraquinone auto-oxidation (AO) process, a complex, energy-intensive, and waste-generating procedure that results in concentrated H_2O_2 , which is often more than what is required for many practical applications (Georgi and Kopinke, 2005). Moreover, the transportation, handling, and storage of concentrated H_2O_2 over long distances introduce potential hazards.

The objective of this study was to explore the use of an electrolytic system for the on-site production of hydrogen peroxide (H_2O_2) to decolorize methylene blue (MB) from wastewater. The goal was to develop a cost-effective and technically feasible alternative to traditional H_2O_2 production methods, such as the energy-intensive anthraquinone auto-oxidation process, which is associated with high costs, excess H_2O_2 production, and safety hazards related to transportation and storage. By utilizing locally sourced activated carbon derived from coconut shells as a catalyst, this study aimed to optimize key process parameters (pH, applied current, and activated carbon weight), to identify the most efficient conditions for dye decolorization, thereby offering a sustainable solution for treating textile wastewater.

Materials and methods

Chemicals

All chemicals were of analytical grade. A 0.05 M Na_2SO_4 electrolytic solution was prepared with deionized water, and its pH was adjusted to 3, 5, 7, 9, and 11 using an Orion Star A111 pH meter, with 0.5 M sulfuric acid (Merck) and 0.5 M sodium hydroxide (Scharlau). Hydrochloric acid served as the activating agent for the coconut shell-activated carbon. Methylene blue dye, the reagent for this study, was procured from B.E. Scientific Chemical Supplier.

Preparation of activated carbon

Coconut shells were collected and thoroughly cleaned with distilled water to remove any dust and impurities. The cleaned shells were then dried in an oven at $110^\circ C$ for 24 hours to eliminate surface moisture. After drying, the shells were

ground to a desired particle size. The resulting material was carbonized in a furnace at 400°C for 30 minutes. Following carbonization, the coconut shell charcoal was crushed with a mortar and pestle and then sieved through a 35-60 mesh screen.

The charcoal was then soaked in a 5% hydrochloric acid solution (500 mL) for 24 hours. After soaking, the charcoal was boiled in deionized water for 60 minutes. The activated carbon was then washed repeatedly with deionized water until the pH of the wash water became neutral. Finally, the charcoal was dried in an oven at 105°C for 24 hours and stored in a sealed container.

Reaction system

All electrolytic experiments were performed in a 250 mL beaker containing 150 mL of 0.05 M Na₂SO₄ solution and 20ppm of MB. The schematic representation of the system is shown in Figure 1. A cell was soaked in the solution. The cell comprises a graphite rod, activated carbon, and an air diffuser. Plastic mesh covers the cell so that the sieved AC would not pass through while bubbling occurs. The anode is also soaked a distance from said cell. The rods are connected to a power supply wherein the current to be applied is adjusted. Electrolysis was performed at different pH levels, applied current, and weight of activated carbon. The time for each run is 90 minutes.

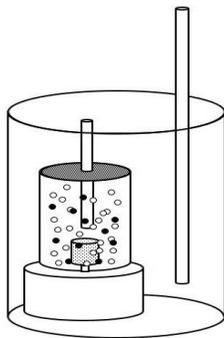


Figure 1. Schematic representation of electrolytic system

Physical and chemical properties of activated carbon

The FT-IR of coconut shell activated carbon was examined by Thermo Nicolet 6700 FTIR Spectrometer. Surface functional groups containing oxygen play a pivotal role in determining the properties of activated carbon. These properties include surface behavior, reactivity, hydrophobicity, surface charges, and the electron density of the graphene layer. Additionally, these oxygen-

containing groups can be utilized for surface modification. Figure 2 illustrates the corresponding peak values and their frequencies. For instance, the peak at 910.2508 cm^{-1} corresponds to the O-H bending mode of carboxylic acids, while the peak at 1488.8 cm^{-1} represents the double bond C=C with a medium weak multi-band. Furthermore, the peak at 2152.203 cm^{-1} attributes to the $\text{-C}\equiv\text{C-}$ stretching mode of alkynes, and the peak at 3671.859 cm^{-1} corresponds to the narrow non-bonded hydroxy group (OH stretch) (Silverstein and Webster, 1997).

The physical surface morphology of coconut shell activated carbon was examined using scanning electron microscopy (SEM). The samples were prepared, and the image was taken using Hitachi S-3400 N Type II. Activated carbon porous material was observed. As shown in Figure 3, the textural characterization of the catalyst determines the surface area, extent microporosity, and pore size distribution. It is shown that after the impregnation of coconut shell using HCl, high porous surface area was developed which means that activated carbon from coconut shell is a good source as a catalyst material (*Nurbazilah et al.*, 2016). In addition, the highly porous texture of the activated carbon is the main criterion of double electric layer, characterized by high capacity and electric exchange. It would be right to make a conclusion that coconut-activated carbon had higher values of specific capacitance due to increasing ion accumulation at the electrode-electrolyte interface with respect to the coconut shell carbon (*Mohd Iqbalidin et al.*, 2013).

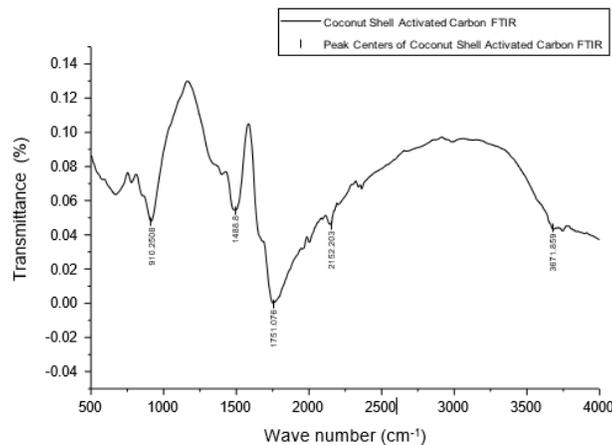


Figure 2. FT-IR analysis of coconut shell activated carbon

Samples were collected from the solution at intervals of 30 minutes. The filtration is determined using the absorption spectra of the filtrate by a Vis

spectrophotometer with atypical quartz cuvettes of 10 mm. The absorbance was measured in the visible region with a wavelength of 681 nm for the samples to determine the decolorization in the MB solution. The calibration curve was measured, and 681 nm was based on the performance. Percentage color removal was calculated as follows:

$$\text{Color Removal, \%} = \frac{C_o - AC_t}{C_o} \times 100 \quad \text{Equation 1}$$

where, C_o and C_t are the concentration of the solution before and after time (t) of electrolysis.

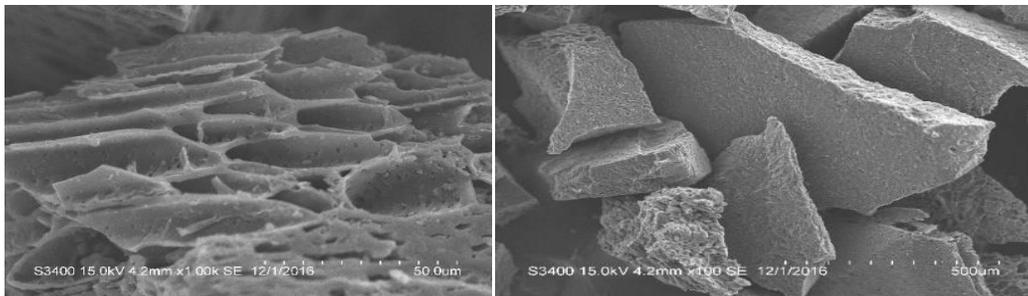


Figure 3. SEM image of coconut shell activated carbon

Results

Optimization of operating variables

The current work was represented Response Surface Methodology (RSM) in the form of three parameters and three levels in CCD (low, mean, and high), where the parameters were the weight of the AC, the pH, and the current density as presented in Table 1. The effects of these three parameters along with their interactive effects were analyzed and discussed.

Table 1. Parameter ranges utilized in optimization studies

Parameters	Low	Mean	High
Wt of AC	0.2	0.6	1
pH	3	7	11
Current	0.1	0.3	0.5

In this study, RSM was applied for the optimization of process variables aimed at enhancing the degradation of methylene blue. This method was

combined with the Central Composite Design (CCD) factorial experimental design to explore the effects of several factors simultaneously. RSM is particularly useful for studying the impact of multiple variables on a particular outcome while minimizing the number of experiments required. Design Expert 7 software was utilized to analyze the experimental data and construct a regression model. For evaluating the effects of various operating conditions on the color removal efficiency of methylene blue, focused on three independent variables: the weight of activated carbon, pH value, and applied current density. A total of 20 experiments based on the CCD matrix were conducted to develop quadratic models, as shown in Table 2.

The predicted vs actual response sqrt (% color removal) and contour plots of the response (%color removal) are shown in Figures 4 and 5.

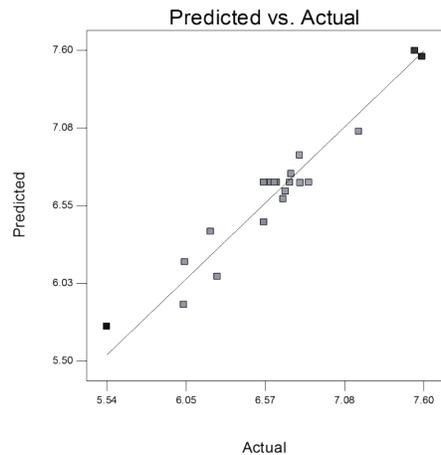


Figure 4. Predicted vs actual values of the responses in color removal

The RSM was used to explore how changes in pH and the amount of activated carbon affect color removal, and we displayed the results as contour plots. Using more activated carbon improves color removal and the effect was stronger when the pH was lower (Figure 5a).

The result indicated that as the current density increased with the decrease of pH value, the color removal of methylene blue increased as shown in Figure 5b. The relationship between current density and weight of activated carbon are evaluated. It is shown that as the current density increased as well as the weight of activated carbon, the removal of methylene blue increased (Figure 5c).

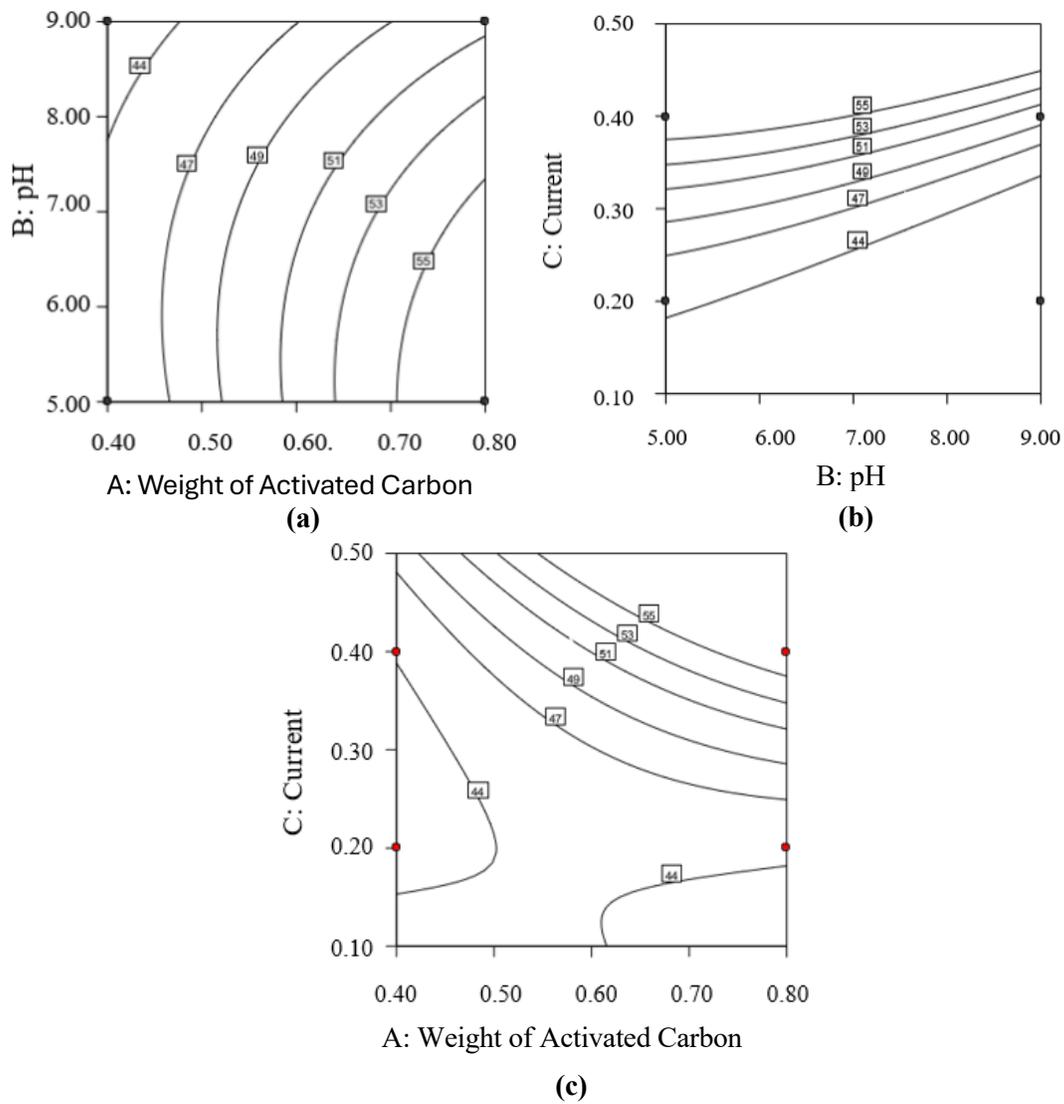


Figure 5. Interaction of Key Variables in Color Removal Using Activated Carbon, (a) Interaction between pH value and weight of activated carbon in color removal, (b) Interaction between current and pH value of activated carbon in color removal, (c) Interaction between current and weight of activated carbon in color removal

Table 2. Optimization results

Run	Wt of AC, g	pH	Current, A	Color removal, %
1	0.6	7	0.3	46.94
2	0.4	5	0.4	44.90
3	1.0	7	0.3	46.16
4	0.8	9	0.4	51.52
5	0.4	9	0.4	43.02
6	0.6	7	0.3	43.88
7	0.6	7	0.3	43.06
8	0.8	9	0.2	36.35
9	0.6	7	0.3	44.10
10	0.6	11	0.3	30.30
11	0.6	7	0.1	38.61
12	0.6	7	0.5	56.71
13	0.6	3	0.3	45.36
14	0.6	7	0.3	45.71
15	0.8	6	0.2	46.02
16	0.6	7	0.3	43.97
17	0.4	5	0.2	44.25
18	0.2	7	0.3	36.33
19	0.4	9	0.2	39.19
20	0.8	5	0.4	57.51

The statistical significance of the second-order regression equation was established with a p-value below 0.0001, affirming that the model is statistically sound. Additionally, the lack of fit was not significant at the 99% confidence level. The regression coefficients and ANOVA findings, with an F-value of 18.96 demonstrating that the model is found to be a valid tool for evaluating dye color removal in the CCD experiment (Table 3).

The model terms with "Prob > F" values below 0.1000 were statistically significant differed in identifying A, C, AC, B², and C² as important factors in the model. Terms with "Prob > F" values above 0.1000 are considered insignificant. The "Lack of Fit F-value" of 3.29 suggested that the lack of fit was not significantly compared to pure error. The model's effectiveness is confirmed by a high R² value of 94.46% for dye color removal. Furthermore, the adjusted R² value of 89.48% supported the model is found to be a good fit for predicting dye removal efficiency.

Table 3. Analysis of variance for the optimization runs

Source	Sum of Squares	df	Mean Square	F value	p-value
Model	725.15	9	80.57	18.96	<0.0001*
A – Wt. of AC	62.63	1	62.63	14.74	0.0033
B – pH	6.36	1	6.36	1.50	0.2494
C – Current	27.67	1	27.67	6.51	0.0288
AB	8.72	1	8.72	2.05	0.1826
AC	61.94	1	61.94	14.57	0.0034
BC	6.66	1	6.66	1.57	0.2392
A2	7.74	1	7.74	1.82	0.2068
B2	48.10	1	48.10	11.32	0.0072
C2	26.98	1	26.98	6.35	0.0304
Residual	42.50	10	4.25		
Lack of Fit	32.60	5	6.52	3.29	0.1083
Pure Error	9.90	5			

*significant

Effects of pH

The decolorization efficiency of methylene blue (MB) using the electro-Fenton process at different initial pH levels is shown in Figure 6. The absorption peak of MB was observed at 681 nm. In this experiment, pH values of 5.0, 7.0, and 9.0 were tested, and after 90 minutes, the highest color removal was achieved at pH 5.0. The solution with pH 9 had the least. Basically, the Fenton and electro-Fenton process was controlled in an acidic solution. However, color removal decreased at higher pH concentrations. An increase in pH led to a lower oxidation potential for hydroxyl radicals (OH), thereby reduced their effectiveness in the decolorization process.

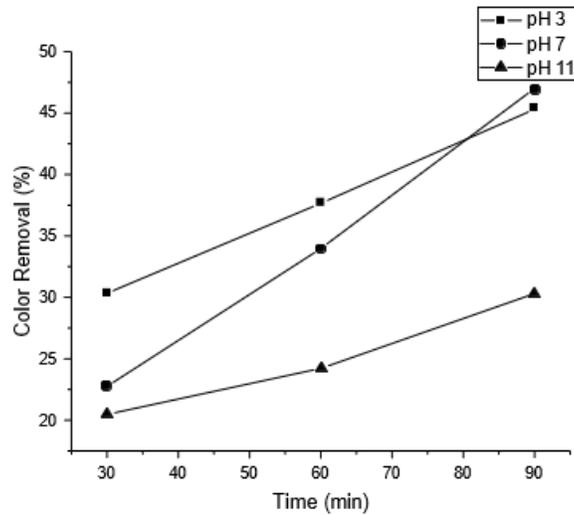


Figure 6. Color removal percentage with the electrolysis time at different pH levels: [MB] = 20 ppm, I = 0.3 A, wt of AC = 0.6g

Effects of applied current

The study investigated the effectiveness of the graphite rod as a cathode material under different current settings ranging from 0.1 to 0.5 A. During the electrolysis of methylene blue at a concentration of 20 mg/L in 0.05 M Na₂SO₄, the hydroxyl radicals generated from water discharge reacted with the oxygen on the anode surface. The outcomes were analyzed using a UV-Vis spectrophotometer. The higher applied current densities resulted in a more rapid and extensive degradation of the dye as illustrated in Figure 7.

Effects of the weight of activated carbon

The effect of coconut shell activated carbon catalyst was evaluated under different weights in grams – 0.2g, 0.4g, 0.6g, 0.8g, and 1g. The results are shown in Figure 8. As the activated carbon increased, the color removal of methylene blue also increased.

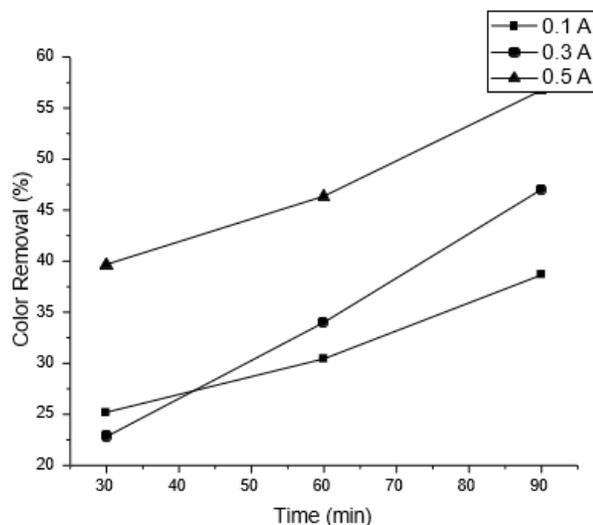


Figure 7. Color removal percentage with the electrolysis time at different applied current: [MB] = 20ppm, pH = 7, wt of AC = 0.6g

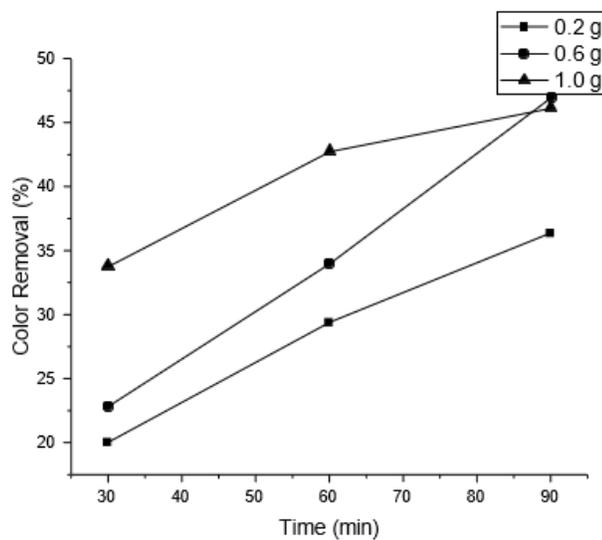


Figure 8. Color removal percentage with the electrolysis time at different weights of AC: [MB] = 20ppm, I = 0.3 A, pH = 7

Electrolysis performance

Experimental results during electrolysis showed that the oxidation process leads to MB color removal reduction from 30.30% to 57.51%. So, color removal percentage was increased by adding Fe^{2+} with electrolysis as shown in Figure 9. Further studies can be carried out with the concentration of H_2O_2 while adding Potassium Titanium Oxalate. Also, this experiment can be held for other dyes or organic pollutants.

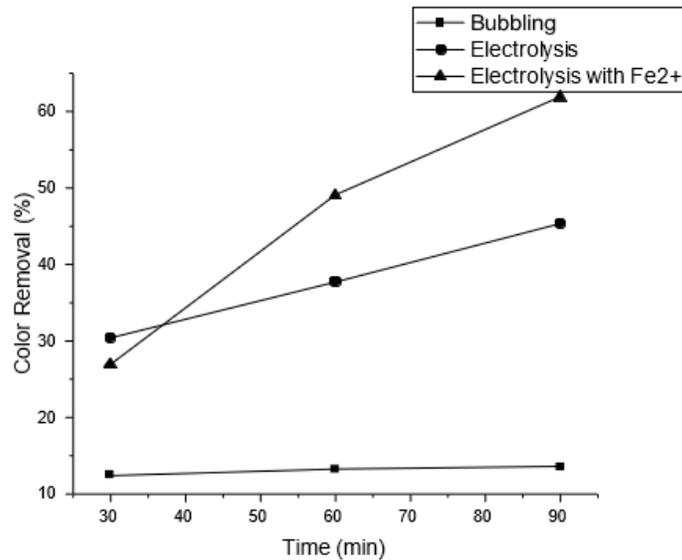


Figure 9. Color removal percentage at different controlled conditions: $[\text{MB}] = 20\text{ppm}$, $I = 0.3\text{ A}$, $\text{wt of AC} = 0.6\text{g}$, $[\text{Fe}^{2+}] = 3\text{ mmol/L.}$, $\text{pH} = 3$

Discussion

The optimum pH is one of the most influential factors on decolorization performance. The best decolorization process performance was at an initial pH of 5, where a color removal of 57.51% was obtained in 90 min. The highest color removal was achieved under the condition of pH 5, applied current of 0.4 A, and 0.8 g of activated carbon.

Thus, the higher the pH showed the lower the color removal. It can be concluded that methylene blue decolorization was much more efficient at lower pH concentrations, which obtained an optimum value of 45% color removal, which would be more favorable for the Fenton reaction (Bañuelos *et al.*, 2014).

The effect of the coconut shell activated carbon catalyst was systematically evaluated under varying weights of 0.2 g, 0.4 g, 0.6 g, 0.8 g, and 1 g which demonstrated its influence on the decolorization of methylene blue. An increase in the amount of activated carbon correlates with enhanced color removal, indicating that the concentration of activated carbon directly influences the efficiency of the system. The activated carbon is shown more active sites available for the oxygen reduction reaction in hydrogen peroxide production. Further, activated carbon had high porosity that influenced the adsorption of organic contaminants and the formation of OH radicals by the catalyst due to its synergistic effect upon electrolysis.

This observed trend suggested that a higher amount of activated carbon provides additional active sites for the oxygen reduction reaction during hydrogen peroxide production (Bañuelos *et al.*, 2014). The presence of these active sites facilitates the catalytic reduction processes, contributing to the observed increase in methylene blue decolorization. Moreover, the high porosity of activated carbon plays a critical role in this process, as it promotes the adsorption of organic contaminants and enhances the formation of hydroxyl (OH) radicals through its synergistic effect under electrolysis conditions (Mohd Iqbalidin *et al.*, 2013). These OH radicals are key intermediates that drive the degradation of methylene blue, underscoring the dual role of activated carbon as both a physical adsorbent and a catalytic surface (Bañuelos *et al.*, 2014; Mohd Iqbalidin *et al.*, 2013).

The decolorization experiments showed the best results which observed at an initial pH of 5, where a color removal rate of 57.51% was achieved within 90 minutes. The optimal conditions for achieving this high level of color removal were identified as pH 5, an applied current of 0.4 A, and 0.8 g of activated carbon. The electrolysis process improved the color removal from 30.30% to 57.51% under these conditions. The addition of Fe²⁺ during electrolysis was significantly increased the percentage of color removal. Future investigations could focus on quantifying H₂O₂ concentration using Potassium Titanium Oxalate and extending the study to include different dyes or organic pollutants. Click or tap here to enter text. The study investigated the performance of the graphite rod as a cathode material under varying current settings (0.1 to 0.5 A) for the electrolysis of methylene blue at a concentration of 20 mg/L in 0.05 M Na₂SO₄. During the process, hydroxyl radicals (•OH) were generated from water reduction at the cathode and subsequently reacted with oxygen species at the anode surface. The results, analyzed using a UV-Vis spectrophotometer, demonstrated that higher applied current densities led to faster and more complete methylene blue degradation.

This can be attributed to increase electron transfer at the cathode with higher current densities, which enhances the production of hydroxyl radicals. These radicals are crucial for the oxidative degradation of methylene blue. Additionally, the reaction of hydroxyl radicals with oxygen on the anode surface further supports this process. The findings align with previous observations that hydroxyl radical generation is key to facilitating dye removal under electrochemical conditions (Bañuelos *et al.*, 2014; Mohd Iqbalidin *et al.*, 2013). Thus, optimizing current density is vital for improving the efficiency of methylene blue degradation through electrolysis. Moreover, the study showed that methylene blue (MB) decolorization was most effective at an initial pH of 5, achieving 57.51% removal within 90 minutes under 0.4 A applied current and 0.8 g of activated carbon. The addition of Fe₂₊ improved the removal rate from 30.30% to 57.51%. Activated carbon's high porosity and active sites facilitated hydroxyl radical formation and MB adsorption, enhancing decolorization. Higher applied current densities promoted faster MB degradation by increasing hydroxyl radical production through improved electron transfer at the cathode. These findings align with Bañuelos *et al.* (2014) and Mohd Iqbalidin *et al.* (2013), highlighting the importance of optimizing current density and other parameters. Future research could explore H₂O₂ quantification and the decolorization of other dyes or organic pollutants.

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