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Optimization Studies on Oxidative Degradation of Methylene Blue Dye using Fenton Reagent

Shreya D.K
PG Student, Department of Environmental Engineering Bapuji Institute of Engineering and Technology, Davanagere, Karnataka, India

Dr. Sumana Y B
Assistant Professor, Department of Civil Engineering Bapuji Institute of Engineering and Technology, Davanagere, Karnataka, India

***Abstract*— The study focuses on optimizing the degradation of methylene blue dye using Fenton's reagent. It aims to improve dye removal efficiency from water by adjusting parameters like pH, Fenton dosage, initial concentration, and contact time. Experiments determined that the best results occur at a pH of 3 with specific Fenton dosage. The findings, supported by UV-Vis spectrophotometric analysis, indicate significant dye reduction. In This research Fenton's reagent's effectiveness and suggests potential for real-world applications. The growing demand for sustainable and efficient urban infrastructure requires the development of optimized sewerage systems that ensure cost-effectiveness, hydraulic efficiency, and environmental sustainability**

***Keywords*— Fenton reagent, Oxidative degradation, Response surface methodology, Box-Behnken Design, Optimization.**

I INTRODUCTION

The textile industry generates substantial waste, particularly from dyes that pose environmental challenges due to their vibrant colors and resistance to breakdown. With urbanization and industrialization, there is an urgent need to effectively remove toxic pollutants from wastewater and contaminated groundwater. Various industries discharge harmful organic compounds into water bodies, leading to contamination. Approximately 15% of global dye production is lost during dyeing and is released as effluent, affecting ecosystems by altering visual characteristics and reducing sunlight penetration, thus hindering photosynthesis. Innovative and affordable wastewater treatment technologies are essential to mitigate these environmental impacts.

OBJECTIVE OF THE STUDY

1. To synthesize Fenton's reagent.
2. To assess the effectiveness of removing the methylene blue dye through a batch process.
3. To assess the quantity of dye absorbed by Fenton's reagent by varying pH, contact time, and the dosage of the Fenton reagent.
4. To investigate the use of the Fenton process for eliminating Methylene Blue Dye.
5. To examine the response surface methodology and Box-Behnken design to maximize the breakdown of Methylene blue dye.

II LITERATURE REVIEW

A. General

The literature review on the optimization of oxidative degradation of methylene blue dye using Fenton's reagent focuses on various studies that explore the effectiveness of this chemical process. It highlights the key parameters influencing the degradation, such as pH, temperature, the concentration of hydrogen peroxide and iron salts, and reaction time. The review summarizes experimental results showing how these factors can optimize the decolorization and mineralization of methylene blue, showcasing Fenton's reagent as an efficient method for wastewater treatment.

B. Literature Studies

Ali Saber et al. (2013) studied wastewater treatment from petroleum refineries using a Fenton-based method with scrap iron powder. They identified optimal parameters (H_2O_2/COD = 10.03, H_2O_2/Fe = 2.66, pH = 3.0) that achieved over 83% COD reduction in 90 minutes, demonstrating the cost-effectiveness of scrap iron as a catalyst.

María del C. Cotto-Maldonado et al. (2017) focused on degrading methylene blue using the photo-Fenton process with various nanostructured catalysts. Their findings showed that iron oxide nanowires (Fe_2O_3) outperform commercial catalysts ($FeCl_2$), highlighting the significance of catalyst morphology for efficiency.

Zamzury S. H. and Zulhaimi H. (2019) improved the Fenton oxidation technique for textile wastewater by analyzing the effects of hydrogen peroxide concentration, ferrous ions, and reaction duration. Their results emphasized the need for precise chemical dosages and reaction timing for optimal color removal.

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Bhavesh T. Moorjani et al. (2021) provided a comprehensive review of the Fenton process in organic wastewater treatment, discussing its mechanisms, benefits, and limitations, alongside optimization techniques and comparisons with other advanced oxidation processes.

Renato Baciocchi et al. (2023) investigated the use of air nanobubbles to enhance the Fenton process in degrading methylene blue. The study found that these nanobubbles improve reaction kinetics and hydroxyl radical production, suggesting they can significantly boost wastewater treatment efficiency.

III METHODOLOGY

A. MATERIALS

The research employed methylene blue, $FeSO_4 \cdot 7H_2O$, H_2O_2, HCl, and NaOH in their unrefined states, and distilled water was used to create all solutions.

a. Stock Solution Preparation

A stock solution of 100 ppm dye was created by weighing 0.1 g of methylene blue and diluting it with distilled water to a final volume of 1000 ml.

To prepare a 100 ml solution, 0.993 g of $FeSO_4 \cdot 7H_2O$ and 4.965 ml of hydrogen peroxide (H_2O_2) were also measured out and diluted with deionized water up to 100 ml

Fig 1(a)Methylene Blue Stock Solution

Fig 1(b) Fenton Reagent

b. Concentration Measurements

A calibration curve is used to determine the concentration of a substance in an unknown sample by comparing it to known concentrations. In this case, a series of dye stock solutions was prepared at concentrations of 20 ppm, 40 ppm, 60 ppm, 80 ppm, and 100 ppm, which will be used to establish calibration points for analysis.

c. pH Adjustments

- The influence of pH levels on the elimination of methylene blue dye was studied utilizing a pH meter with values ranging from 2.0 to 4.0. The optimal pH for decolorizing the observed dyes was found to lie between 3 and 4.
- The solutions pH was modified using NaOH and HCl. This pH optimization was conducted at 30°C, with each reaction set to a volume of 100 ml.
- The solution was stirred, and samples were taken at various intervals for measurement using a UV-Vis spectrophotometer. The oxidative degradation was assessed using a specific equation.

Fig. 2: pH meter

d. Samples Agitation

After weighing and adding the Fenton dosage to the solutions, the conical flasks were promptly placed in the shaker. A rotational speed of 50 RPM was set, with varying agitation times maintained for different samples to assess various parameters.

Fig 3 Agitation of the samples in the shaker at 50 RPM

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After the agitation is completed, the solution is collected and analyzed using a UV spectrophotometer, with each solution varying in Fenton dosage, contact time, and concentration.

Fig 4 Samples after agitation

e. Spectrophotometer Calibration For Methylene Blue

- Calibration for methylene blue were created at various concentrations, designated as 20 PPM, 40 PPM, 60 PPM, 80 PPM, and 100 PPM.
- These solutions were utilized to generate a standard graph.
- Absorbance measurements were recorded, and the resulting standard graph is presented below.
- Absorbance values were taken at 664 nm, corresponding to the peak absorbance.

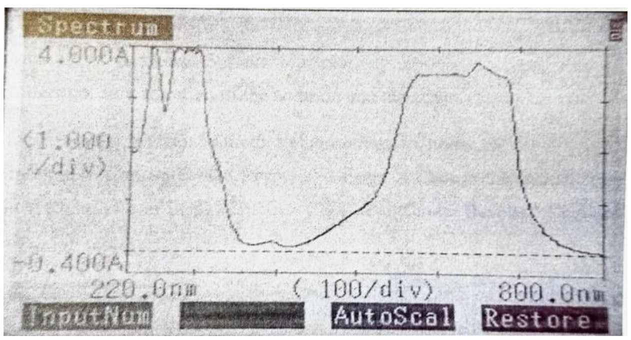

Fig 5 The spectrum displays the highest absorbance at 664 nm for MB dye.

Absorbance V/S Concentration

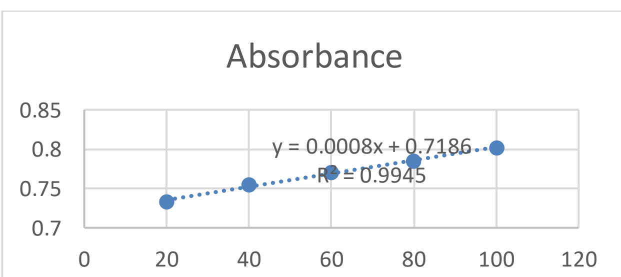

Fig 6 Absorbance V/S Concentration graph

f. Ultraviolet And Visible Absorption Spectroscopy (Uv-Vis)

Ultraviolet and visible absorption spectroscopy (UV-Vis) analyzes the decrease in light intensity as it passes through or reflects off a sample. It can be performed at specific or broad wavelengths, effectively promoting valence electrons to higher energy levels. This technique is useful for studying inorganic complexes or dissolved molecules and is limited in some applications.

The Beer-Lambert Law ($A = \varepsilon * L * C$) relates absorbance (A) to the absorptivity coefficient (ε), path length (L), and concentration (C). The UV-Vis range (400 to 750 nm) helps characterize materials like pigments, coatings, and filters, while also allowing for the study of their optical and electronic properties. A wavelength of 664 nm was established on the spectrophotometer to analyze the supernatant solutions. Concentration levels were assessed using Concentration levels were determined using a standard calibration curve, and necessary dilutions were carried out.

Following this, the quantity absorbed by the adsorbent was calculated by back-referencing the concentration of the supernatant. Furthermore, the proportion of adsorption was also assessed.

Fig 7. UV- Spectroscopy

g. Box-Behnken Design Operation

Before using the Box-Behnken design, the traditional method involved adjusting parameters like contact time (5 to 30 minutes), Fenton dosage (2 to 10 ml), and Methylene Blue concentrations (20 to 100 ppm) individually. Experiments were structured using Minitab software following the Box-Behnken design based on Response Surface Methodology, selecting various ranges for the parameters. The steps included opening Minitab, navigating to DOE, selecting Response Surface Method, and configuring the Box-Behnken design.

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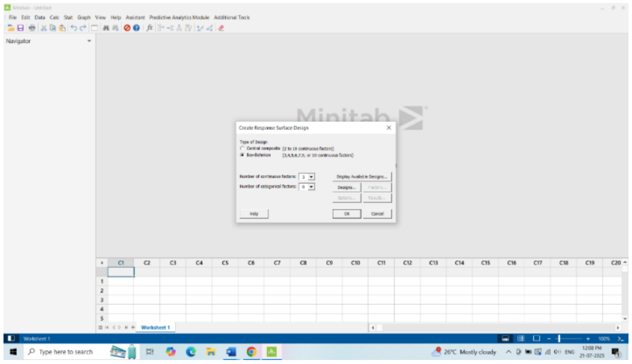

Fig 8. Selection of no. of factors in Minitab Software based on Box-Behnken Design

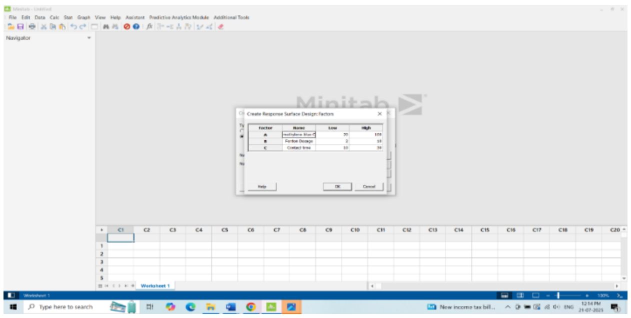

Fig 9. Response Surface design for various combination of samples

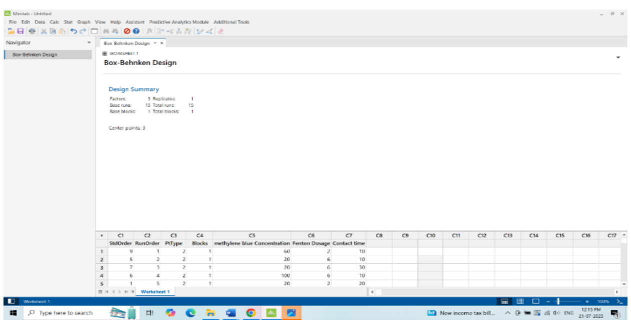

Fig 10. Choosing Ranges for Parameters in a Response Surface Design

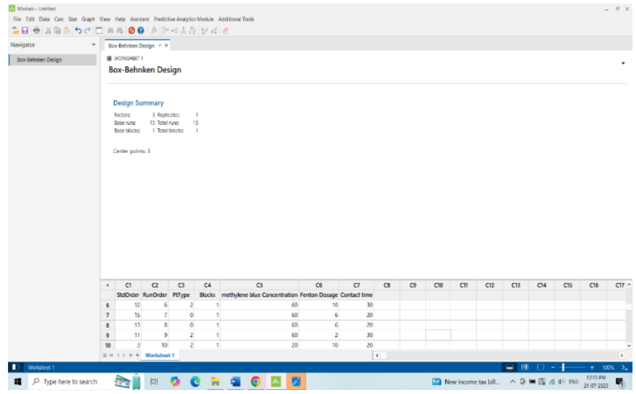

Fig 11. Choosing Ranges for Parameters in a Response Surface Design

BOX-BEHNKEN DESIGN

Table 1(a) Design Summary of Box-Behnken Design

Centre of points: 3

Factors: 3	Replicates: 1
Base runs: 15	Total run: 15
Base blocks: 1	Total blocks: 1

Table 1(b) Selection of effective ranges of parameters

Factors	**Units**	**Minimum value**	**Maximum value**
Dosage	Gram	2	10
Concentration	PPM	20	100
Contact time	Minutes	5	30

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Table 2 .Box-Behnken Approach for the Specified Parameter Ranges

Run order	Fenton Dosage	Concentration	Contact period
1	2	60	10
2	6	20	10
3	6	20	30
4	6	100	10
5	2	20	20
6	10	60	30
7	6	60	20
8	6	60	20
9	10	60	30
10	2	20	20
11	6	60	20
12	2	100	20
13	10	60	10
14	10	100	20
15	6	100	30

Calculations:

Percentage removal (%) = $(C_i-C_f) / C_i \times 100$

Where,

Ci = Initial concentration (mg/L) at any instant of time

Cf = Final concentration (mg/L) at any instant of time

IV RESULTS

1.VARIATION IN FENTON DOSAGE

Sl.no	Fenton Dosage	Methylene blue conc	pH	% Removal
1	2	100	3	31.5
2	4	100	3	45.4
3	6	100	3	58.8
4	8	100	3	72.9
5	10	100	3	79.5

Table 3. Impact of Fenton dosage

To assess the effect of varying Fenton dosage on removal efficiency, a batch adsorption method was employed. Different volumes of Fenton were added to the solution, including 2ml, 4ml, 6ml, 8ml, and 10ml concentrations. The results are illustrated below. The efficiency of removal gradually improved from 2ml to 10ml, ultimately reaching 79.5% at the 10ml dosage.

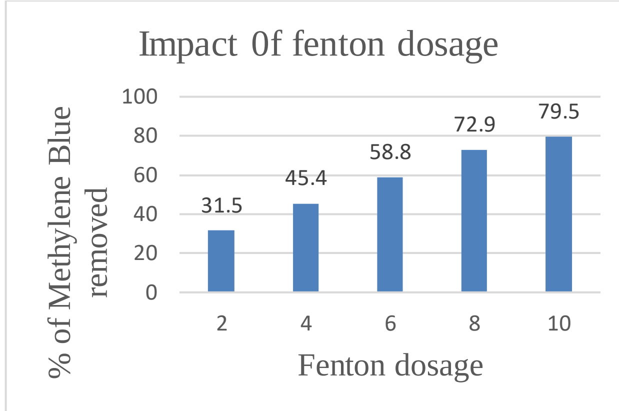

Fig 12.Graph showing percentage removal w.r.t Fenton Dosage

2.VARIATION IN INITIAL CONCENTRATION

Sl. No	Fenton Dosage	Methylene blue concentration	Contact time	% Removal
1	2	20	30	84.4
2	2	40	30	70.8
3	2	60	30	60.2
4	2	80	30	48.7
5	2	100	30	36.1

Table 4. Impact of Initial Concentration

To examine how initial concentration affects removal effectiveness, batch of experiments were conducted. Various concentrations of 20ppm, 40ppm, 60ppm, 80ppm, and 100ppm were prepared for the adsorption tests. The removal efficiency was 84.4% at 20ppm and consistently decreased with increasing concentration, dropping to 36.1% at 100ppm.

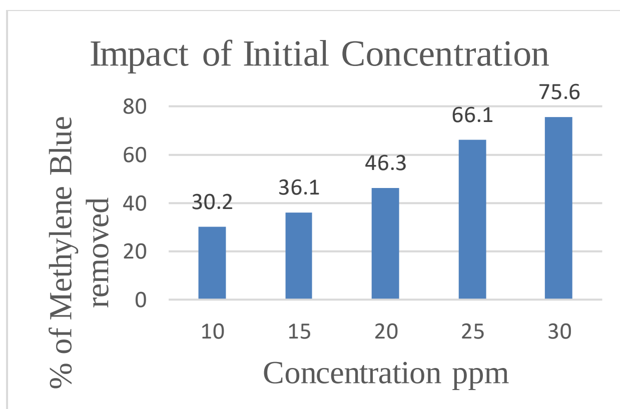

Fig 13.Graph showing percentage removal w.r.t Initial Concentration

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3. IMPACT OF CONTACT TIME

Sl.no	Fenton dosage	Methylene blue conc	Contact time	% Removal
1	2	100	10	30.2
2	2	100	15	36.1
3	2	100	20	46.3
4	2	100	25	66.1
5	2	100	30	75.6

Table 5. Impact of Contact Time

To investigate how varying contact time influences the percentage removal, batch of absorption methodology was employed. Contact times chosen were 10 minutes, 15 minutes, 20 minutes, 25 minutes, and 30 minutes. The concentration of the solution was maintained at a constant level at 100 ppm, and 2 ml of Fenton's reagent was added to the samples. The findings from the experiment are illustrated in the plot below. At the 10-minute mark, the methylene blue concentration adsorption was recorded at 30.2%, which rose to 75.6% after 30 minutes.
It was noted that there was an adsorption rate during the initial phase, continuing steadily up to 30 minutes.

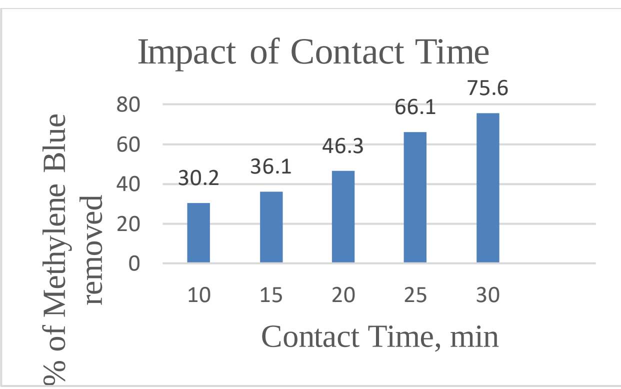

Fig 14.Graph showing percentage removal w.r.t Contact time.

V CONCLUSION

The use of the Fenton reagent, which combines hydrogen peroxide and ferrous iron, has emerged as an advanced method for enhancing the oxidative at various factors, including pH, initial dye concentration, the amount of breakdown of methylene blue dye in wastewater treatment containing dyes. Research commonly demonstrates that of Fe^{2+}, the dosage of H_2O_2, and reaction duration, significantly influence the effectiveness of the degradation process.

A. Scope For Future Studies

Although the Fenton process is well-defined, there are numerous fields ripe for investigation and enhancement to boost its practical applicability, cost-effectiveness, and environmental sustainability. Future research can focus on the following avenues:

- Utilization of Modified Fenton Reagents
- Integration of Processes and Hybrid Systems
- Optimization through Modeling and AI Applications
- Practical Applications in Wastewater Treatment
- Evaluation of Environmental Impact and Toxicity
- Advancement of Electro-Fenton Technologies

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