Published by :
https://www.ijert.org/
An International Peer-Reviewed Journal

International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 14 Issue 11 , November - 2025

Degradation of Acid Blue 9 using Hydrogen Peroxide

Shane DCosta, Aryan Chaure, Vaibhav Bhuwaniya, Abhinandan Daga, Vaibhavi Chavan
Student Vishwakarma Institute of Technology, Pune
Gayatri Gawande
Professor Vishwakarma Institute of Technology, Pune

***Abstract* - The project investigates the removal of Acid Blue 9 from wastewater through simple oxidation using H_2O_2. The influence of pH, Initial dye concentration, H_2O_2 concentration and time was investigated to find the best parameters for degradation of the dye. To carry out the experiment, synthetic wastewater samples were created using the dye and then degradation was carried out on them. Then, to quantify the degradation, COD analysis was carried out. Highest Degradation was obtained at 2 pH with a H_2O_2 concentration of 3.5 ml/L at 2 hours which was 84.96 %. There were no significant changes in COD value between 3.5 ml/L and 4 ml/L H2O2 concentration and beyond 2 hours contact time. Further research can be done using AOP methods.**

***Keywords* – (Acid Blue 9, H_2O_2, pH, concentration, time)**

1. INTRODUCTION

In recent years, synthetic dyes have become essential components in various industries, particularly textiles, food processing, cosmetics, and paper manufacturing [1]. Among these, Acid Blue 9 is a commonly used dye known for its vibrant color and stability. However, dyes like Acid Blue 9 also present environmental challenges due to their resistance to natural degradation processes[2]. When discharged untreated into water bodies, they not only disrupt the aesthetic quality of water but also pose serious risks to aquatic ecosystems and potentially to human health. This resilience is due to their complex molecular structures, which make conventional wastewater treatments less effective at achieving complete dye removal[3].

In recent years, wastewater treatment systems have been improved and more advanced methods have been developed. In the past, it was very difficult to treat certain wastewater pollutants but with research in the field, many of the pollutants today can be treated. However, some pollutants which are classified as ECs (Emerging Contaminants) continue to be difficult to treat[4]. These compounds can significantly damage the environment as well as the use of the compounds has grown in recent years, Acid Blue 9 being one of them. Acid Blue 9 gained prominence in industries in the last few years and therefore there is not much research into treating it[5]. Therefore, we chose it for our project with our goal being to treat it to the highest degree possible.

Due to its persistence in aquatic environments and limited degradation by conventional methods, there is an urgent need to explore alternative, eco-friendly treatment approaches. So, to combat this, we are going to carry out oxidation of the dye using H_2O_2. In oxidation, the complex molecular structure of the dye is broken down into simpler molecules using H_2O_2 as the oxidizing agent[6]. When H_2O_2 is added to water, it dissociates into H_2O and O_2 as given below :

2H2O2 → 2H2O + O2

and therefore it is environmentally friendly[7]. The O_2 radicals come into contact with the dye in the dye solution and then degrade it into simpler molecules which are less/not harmful to the environment. Only simple H_2O_2 has been used in order to minimize the environmental impact[8].

For our project, we first have created sample dye solutions using a fixed concentration of dye in water. Then, we carried out oxidation of the dye solutions using a fixed amount of H_2O_2. After oxidation, Cod analysis is carried out of the dye samples in order to determine the degradation percentage of the samples. The main purpose of our project is to vary and optimize parameters in each run and then find the best set of parameters for the process. The parameters that were varied were pH, H_2O_2 concentration, Dye concentration and degradation time. After each run, COD analysis is carried out and degradation percentage is obtained. After every run in a parameter is carried out, degradation percentage of each run is compared and the best one is selected. Then that specific parameter is fixed and another parameter is varied. In this way, best set of parameters are obtained.

To quantify the degradation amount, COD analysis is carried out. COD stands for Chemical Oxygen Demand. It indicates the amount of oxygen utilized by reactions in a solution[9]. It is commonly expressed as mass of oxygen consumed over a volume of solution which in SI units is milligrams per litre (mg/L). Most common application of COD is to determine the amount of organics or oxidizable pollutants in a body of water (lakes, rivers) or wastewater[9].

This study highlights how varying parameters of a process can adversely affect the results. By gathering data and varying the parameters after each set of runs, we can identify the best set of parameters and therefore optimize the process for better efficiency. Further, for oxidation, only simple H_2O_2 is used without additional reagents to minimize costs and environmental impact.

The maximum degradation percentage obtained was 84.96 %. Future research can be done using advanced methods like Advanced Oxidation Process (AOP). Using AOP, degradation percentage can be increased but it also increases the costs and the complexity of the process.

2. METHODOLOGY

2.1. Chemicals Used:

The chemicals used in this study include Acid Blue 9 ($C_{37}H_{34}N_2Na_2O_9S_3$), Hydrogen Peroxide ($H_2O_2$), Distilled Water ($H_2O$), Potassium Dichromate ($K_2Cr_2O_7$), Concentrated Sulphuric Acid (H_2SO_4), Silver Sulphate (Ag_2SO_4), Ferrous Ammonium Sulphate ($(NH_4)_2SO_4{\cdot}Fe(SO_4){\cdot}6H_2O$), and Ferroin Indicator ($[Fe(C_{12}C_8N_2)_3]SO_4$). All chemicals used were of analytical grade.

Acid Blue 9 Dye was supplied by Sharad Agencies, Pune. The other chemicals which are Hydrogen Peroxide, Potassium Dichromate, etc were supplied by Loba Chemie. The structure of the dye is given in Fig 1. H_2O_2 (30 %) was used. All solutions were prepared using distilled water. A 1 L dye solution was prepared, from which 200 mL samples were taken for each experiment. Initial pH was varied using NaOH and H_2SO4. Amount of NaOH and H_2SO_4 was negligible compared to the total reactor volume. Experiments were carried out at room temperature (27 °C). All instruments were cleaned before use. Due to disintegration, new $FeSO_4$ Solution was created everyday and old solution was disposed[9]. After each experiment, COD was carried out using open reflux method.

2.2. Acid Blue 9 Properties and structure:

Acid Blue 9 is a solid compound with a melting point of 283°C and an estimated density of 1.1666 g/cm^3. It has a vapor pressure of 0 Pa at 25°C and a refractive index of approximately 1.6000. The dye should be stored in an amber vial under refrigerated conditions to maintain stability, as it is light-sensitive. It is dark blue to very dark purple in color and exhibits slight solubility in DMSO, ethyl acetate, and methanol, while being highly soluble in water with a solubility of 1000 mL/L at 24.3°C. Its logP value is -3 at 23°C, indicating high hydrophilicity.

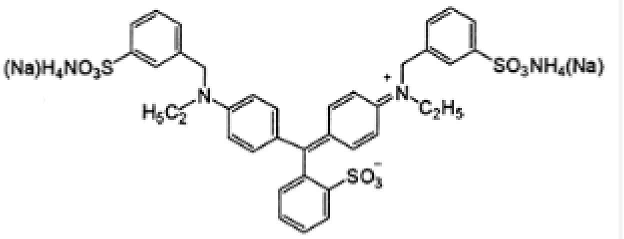

Fig.1. Structure of Acid Blue 9

2.3. COD Reagents Preparation:

a. Standard Potassium Dichromate Reagent (Digestion Solution) –

To prepare the standard potassium dichromate reagent, 4.913 g of potassium dichromate was first added to a clean beaker. Next, 167 mL of concentrated sulphuric acid was measured using a clean measuring cylinder and transferred to the beaker, allowing the potassium dichromate to dissolve completely. After the solution was allowed to cool to room temperature, it was transferred to a 1000 mL standard volumetric flask and the volume was made up to the mark using distilled water.

b. Sulphuric Acid Reagent
(Catalyst Solution) –

To prepare the sulphuric acid catalyst solution, 5.5 g of silver sulphate crystals was transferred to a 1000 mL beaker. Subsequently, 500 mL of concentrated sulphuric acid was added to the beaker, and the mixture was allowed to dissolve over a period of two days.

c. Standard Ferrous Ammonium Sulphate Solution –

To prepare the standard ferrous ammonium sulphate solution, 39.2 g of ferrous ammonium sulphate was weighed and transferred to a 1000 mL standard volumetric flask. Distilled water was then added to bring the volume up to the 1000 mL mark, and the contents were mixed until fully dissolved.

1.4. Oxidation Process:

To begin the experimental process, 1000 mL of dye solution was prepared using a fixed concentration of Acid Blue 9 dissolved in distilled water. From this stock solution, 200 mL samples were used for each individual experiment. A predetermined amount of hydrogen peroxide (H_2O_2) was then added to each sample. The mixture was allowed to stir on a magnetic stirrer for a duration of 1 to 2 hours. Following the reaction period, the samples were analyzed using the COD (Chemical Oxygen Demand) process.

1.5. COD Process

To carry out the COD analysis, 2.5 mL of the dye sample was first added to a COD digestor vial. This was followed by the addition of 1.5 mL of potassium dichromate reagent and 3.5 mL of the catalyst solution. The vial was then securely capped and placed on the digestor, where it was heated at 150°C for a duration of two hours. After the digestion period, the digestor automatically shut off, and the vial was allowed to cool to room temperature.

For the titration step, ferrous ammonium sulphate solution was added to a burette, adjusted to zero, and fixed to a stand. The cooled contents of the vial were transferred to a clean conical flask, and a few drops of ferroin indicator were added, turning the solution bluish green. The solution was then titrated against the ferrous ammonium sulphate until a reddish-brown endpoint was observed. The volume of ferrous ammonium sulphate used during titration was recorded.

The COD value was then calculated using the following formula:

$$\text{COD} = ((A - B) \times N \times 8 \times 1000) / \text{Volume of sample taken}$$

where A is the volume of titrant used for the blank, B is the volume used for the sample, and N is the normality of ferrous ammonium sulphate.

Finally, the percentage degradation of the dye was determined based on the difference in COD values before and after treatment.

1.6. Experiment Procedure:

The experimental process began with the determination of the COD of a blank sample. This was followed by measuring the initial COD of a 30 ppm dye solution without the addition of hydrogen peroxide. Subsequently, the dye solution at 30 ppm was treated with 0.5 mL of hydrogen peroxide at varying pH levels—specifically 11, 9, 7, 5, 3, and 2—and stirred for one hour, after which COD analysis was performed for each condition. The percentage degradation was calculated using the formula: $((\text{Initial COD} - \text{Final COD}) / \text{Initial COD}) \times 100$.

The pH value at which the highest degradation percentage was achieved was selected as the optimal condition and fixed for subsequent experiments. With pH held constant, the concentration of hydrogen peroxide was varied from 0.5 to 4.0 mL/L in 0.5 mL/L increments, followed by COD analysis to evaluate performance. The H_2O_2 concentration that yielded the highest degradation was identified as optimal and fixed, after which the dye concentration was varied across 30 ppm, 50 ppm, and 100 ppm. COD analysis was again conducted for each concentration.

Finally, with the optimized pH, H_2O_2 dose, and dye concentration established, the degradation time was varied among 1 hour, 1.5 hours, and 2 hours. COD values were measured at each interval to assess the effect of contact time on dye degradation efficiency.

3. RESULTS AND DISCUSSION

1.1. Effect of pH on Degradation Efficiency –

The percentage degradation of Acid Blue 9 at 11,9,7,5,3,2 pH was 14.63, 20.325, 26.82, 30.08, 35.36, 39.02. As we can observe, the highest degradation percentage was obtained at an acidic pH of 2. This happens due to a couple of reasons one of them being that at acidic pH, H_2O_2 decomposes more effectively into oxygen and therefore degradation is more efficient. Also, many dyes (including Acid Blue 9) may undergo structural changes at different pH values. In acidic pH, the dye molecules might be more susceptible to oxidation due to changes in ionization or conformation, making them easier to break down.

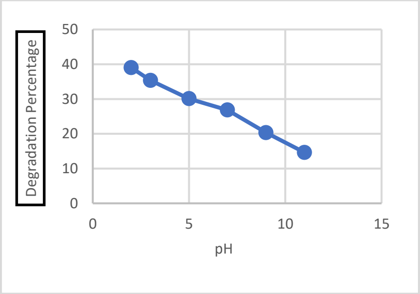

Fig.2. Effect of pH on Degradation efficiency

1.2. Effect of H2O2 concentration on degradation efficiency –

After fixing the pH at 2, experiments were carried out at varying concentrations of H_2O_2 at 1.0, 1.5, 2, 2.5, 3, 3.5, 4 ml/L. The degradation percentage obtained at the above concentrations were 44.3, 51.21, 58.94, 63.41, 65.44, 67.47, 67.47. There was no change in the values obtained at 3.5 and 4 ml/L H2O2 concentration. From this, we can infer that the highest degradation is obtained at 3.5 ml/L H2O2. This happens because when H_2O_2 concentration is increased, we get more reactive oxygen species and therefore, more degradation takes place. But, after a certain concentration, there is no significant increase in the degradation.

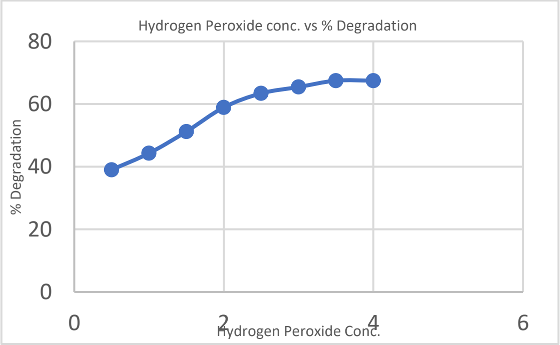

Fig.3. Effect of H_2O_2 concentration on Degradation efficiency

1.3. Effect of initial dye concentration on degradation efficiency –

After fixing pH at2 and H_2O_2 concentration at 3.5 ml/L H_2O_2, dye concentration was changed from 30 ppm to 50 ppm and then 100 ppm. The degradation obtained at each value is 67.47, 47.15 and 2.43. This shows that degradation varies linearly with initial dye concentration as observed on fig.4.

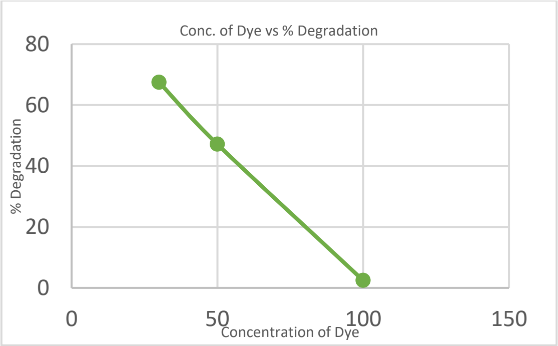

Fig.4. % Degradation vs Initial Dye Concentration

1.4. Effect of degradation time on degradation efficiency –

After the above parameters were fixed, time of degradation was changed from 1 hour to 1.5 hour and then to 2 hours. Degradation obtained was 67.47, 77.64 and then, 84.96. This is because, the more the time is increased, the more time oxidation of the dye occurs and therefore the degradation increases. But the time can only be increased up to a certain level. After 2 hours, not much difference in degradation is observed. This is because after 2 hours, most of the oxidation takes place and therefore, not much reaction happens. Also, increasing time after a certain level reduces the efficiency of the process as a whole.

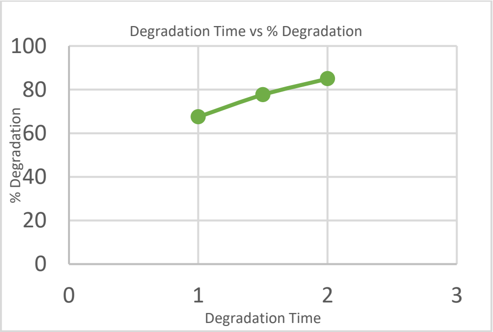

Fig.5. Degradation time vs % Degradation

4. CONCLUSION

In the above study, synthetic solution created using Acid Blue 9 Dye was degraded using oxidation process via H_2O_2 without the use of any catalysts or advanced reagents. The influence of various process parameters such as pH, H_2O_2 concentration, Initial Dye concentration, and degradation time was evaluated. Based on the experimental findings, the following conclusion can be drawn –

Acid Blue 9 was effectively degraded using hydrogen peroxide, achieving a maximum degradation efficiency of 84.96% under optimized conditions. The degradation process was significantly enhanced under acidic conditions, with the highest efficiency observed at pH 2, likely due to increased generation of reactive oxygen species from hydrogen peroxide. An increase in H_2O_2 concentration up to 3.5 mL/L improved degradation, beyond which no substantial enhancement was observed, indicating this as the optimal dosage. Conversely, increasing the initial dye concentration led to a decrease in degradation efficiency, demonstrating an inverse relationship between dye load and treatment effectiveness. Contact time also played a critical role; degradation improved up to 2 hours, after which further increases in duration produced minimal additional benefit.

The process is straightforward, cost-effective, and environmentally benign, as it relies solely on hydrogen peroxide without the use of external catalysts or UV irradiation. These results suggest that the method offers promising potential for the treatment of dye-containing wastewater. Future studies could explore the incorporation of advanced oxidation processes (AOPs) to further enhance degradation performance.

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