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Decolourization of Rhodamine B from aqueous solution by electrochemical oxidation using graphite electrodes

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ABSTRACT

This study investigated the colour removal efficiency of Rhodamine B (RhB) by electrochemical oxidation. The influences of various operating variables such as current density (from 5 to 25 mA/cm^2), initial concentration of dye solution (from 50 to 250 mg/L), initial pH of solution (3, 5, 7, 9), the stirrer speed (150–300 rpm) and the concentration of electrolytes (0.025–0.10 M NaCl) on removal efficiency were explored in a batch mode treatment to achieve a higher removal capacity. The results obtained with synthetic wastewater revealed that the most effective removal of dye could be achieved when the pH was kept neutral. The results showed that the colour removal increases with the increase in applied current density, NaCl concentration, while it was found to decrease with the increase in initial dye concentration. The complete (100%) colour removal has been achieved under the optimal experimental conditions such as NaCl concentration of 0.05 M, applied current density of 15 mA/cm^2, initial pH 7.0, stirrer speed of 200 rpm and electrolysis time of 40 min. The result of UV–vis spectrum analysis confirms the removal of colour from RhB aqueous solution at the short treatment time. In addition, the increase of current intensity, in the range of 0.6–2.0 A, enhanced the treatment rate. The method was found to be highly efficient and relatively fast compared to conventional treatment techniques.

Keywords: Rhodamine B; Electrochemical treatment; Colour removal; Graphite anode

1. Introduction

The widespread disposal of industrial wastewater containing organic dyes onto land and water bodies has led to serious contamination in many countries worldwide because of their toxicity and threat for human life and the environment [1,2]. Among the wet processing industries, the textile industry generates a huge volume of wastewater during sizing, scouring and bleaching, dyeing and finishing processes. Wastewaters from textile dyeing and finishing industries are significant source of environmental pollution because their high concentration of organic compounds can seriously damage the aqueous

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ecosystems. More than 10,000 dyes have been widely used in textile, paper, rubber, plastics, leather, cosmetic, pharmaceutical and food industries, which generated huge volume of wastewater every year [3]. Dyes are normally very large aromatic molecules consisting of many linked rings [4]. There are many processes to remove dyes from coloured effluents such as adsorption, precipitation, chemical degradation, photodegradation and biodegradation [3–10]. Conventional biological treatment processes under anaerobic conditions are ineffective for the treatment of synthetic dyes and the high salinity of wastewater containing dyes because azo dyes are readily reduced to potentially hazardous aromatic amine. Adsorption and coagulation practices are also applied to treat dyes in wastewater, which always result in secondary pollution [2]. Traditional treatment technologies in practise, for removal of dye wastewater fail to meet strict discharge standards. Therefore, an efficient method to treat wastewater containing dye is an urgent need to comply the discharge standards [11].

Electrochemical oxidation is applied for remediation of pollutants in recent years. Electron has the main role in this technique; hence, it is a clean and eco-friendly process. Electrochemical oxidation has advantages of no chemicals involvement in the treatment process, no sludge generation, besides cost-effectiveness and requirement of small area. Other advantages of electrochemical oxidation are: (1) can be operated at room temperature and pressure, (2) high efficiency and (3) capability to combine with other treatment processes [7,12].

The present work was designed to investigate the colour removal efficiencies of aqueous RhB solution using graphite electrode under various operating conditions in an undivided electrochemical reactor. The operating variables that affect the removal of colour such as the applied current density, the pH of solution, the stirrer speed, the concentration of RhB dye and electrolytes were investigated.

2. Materials and methods

2.1. Materials

The dye RhB (commercial grade) was obtained from Merck (Mumbai, India). The working solution of required concentration was prepared by diluting the stock solution and adding appropriate concentration of supporting electrolyte. The pH of the aqueous solutions was adjusted by dilute H_2SO_4 or NaOH solutions. The other chemicals used were of analytical grade and procured from M/s Merck, Mumbai, India. Graphite electrode, supplied by M/s. Carbone Lorraine (Chennai, India) was used as anode and cathode. The electrical resistivity of graphite sheets was 0.001 Ωcm. The graphite sheet assays 99.8% C, <150 mg/L of Fe and <30 mg/L of Cl [13]. An active total anodic surface area of graphite sheets was 102 cm^2. Double-distilled water was used in all the experiments.

2.2. Electrochemical cell

The experiments were conducted in an undivided cell of 650 mL capacity. The graphite plate with a thickness of 0.2 cm was used as an electrode. Four graphite plates (two each for anode and cathode) were internally looped to form an anode and cathode assembly. The gaps between the plates were maintained at 6 mm to minimize the ohmic losses. Graphite was used as anode in view of the fact that O_2 overpotential is high on graphite while Cl_2 overpotential is low, this would increase current efficiency of Cl_2 evolution [7]. Electric power was supplied by a regulated DC power supply obtained from M/s. Mighty Electronics Equipment Corporation Pvt. Ltd, Coimbatore, India. The entire electrode assembly was placed on non-conducting wedges fixed to the bottom plate of the electrochemical cell [14].

2.3. Experimental methods

For each experiment, 500 mL of the RhB dye solution was taken into the electrochemical cell and the electrodes were connected to the respective anode and cathode leads of the DC power supply and energized for a required duration at a fixed current. All the experiments were performed at room temperature. During the experiment, the solution was placed on a magnetic stirrer for continuous mixing at 200 rpm in order to ensure the mass transfer efficiency. At different time intervals during the reaction, samples were collected and determined for percentage removal of colour.

2.4. Analytical techniques

The colour removal was chosen as the parameter for evaluating the effect of electrochemical treatment and measured by spectra photometer (UV–1700 Pharma Spec, Shimadzu, Japan) at 553.8 nm. The pH of the synthetic wastewater was adjusted by 0.1 M hydrochloric acid (Merck, India) or 0.1 M sodium hydroxide (Merck, India) and measured by a Elico pH meter (India). Spectrums of samples were recorded by an UV–vis spectrophotometer (UV–1700 Pharma Spec, Shimadzu, Japan) and the maximum visible absorption wavelength was measured. Colour removal efficiency is determined according to Eq. (1):

$$\text{Colour removal efficiency } (\%) = \left(\frac{A_o - A}{A_o}\right) \times 100 \qquad (1)$$

where A_o and A are the light absorbances of dye before and after electrochemical treatment, respectively. In this study, each treatment was repeated twice and the absorbance measurement of each sample was repeated three times. The standard errors were all within 10% of the mean values.

3. Results and discussion

3.1. Effect of initial pH on colour removal

Initial pH is an important factor that influences the degradation reaction. In order to study the pH influence, the experiments were conducted at four different initial pH values (3, 5, 7 and 9) and pH of the solution was adjusted using sulphuric acid (0.1 N H_2SO_4) or sodium hydroxide (0.1 N NaOH). During the treatment process, the current density was maintained at 15 mA/cm^2, electrolysis time of 30 min, stirrer speed of 200 rpm and 0.05 M of NaCl as the supporting electrolyte. Fig. 1 shows the influence of initial pH on the percentage colour removal by electrochemical oxidation within 30 min of the reaction.

It is clear that varying the initial pH strongly affected electrochemical degradation efficiency on the colour removal reached 97.72% at pH 7.0 within 10 min, while lower colour removal was observed at higher pH. This is due to the oxidation and hydrolysis of chlorine yields hypochlorous acid (HOCl) or the hypochloride ion (OCl^-) depending on the solution pH. At pH 7, it was necessary to increase contact time to longer than 10 min in order to achieve higher than 93.9% removal. On the other hand, a low pH solution will corrode the electrode and inhibit the activity of electrode, even hinder the proceeding of degradation reaction [15]. Yavuz and Kaporal [16] have reported that electrochemical oxidation without initial pH adjustment (initial pH around 7.0) was more effective in removing phenol, compared to pH 3.0 and pH 11. Due to high removal of colour, initial pH 7.0 was chosen for the electrochemical oxidation of aqueous RhB solution for all the experiments.

3.2. Effect of supporting electrolyte concentration on colour removal

The electro conductivity of solution is another important factor in the degradation reaction. High ionic strength leads to faster electron transport and better degradation rate [15], so the effect of electrolyte on the degradation of RhB was investigated. Solutions with various ionic strengths were obtained by preparing various concentrations of NaCl aqueous solution in this study. Fig. 2 presents the effect of NaCl concentration under fixed current density and dye concentration.

It is clear that increasing chloride concentration increases the percentage of colour removal. The reason is that increasing NaCl concentration lowers the discharge potential of Cl_2 according to the Nernst equation [17], and therefore, more current is consumed

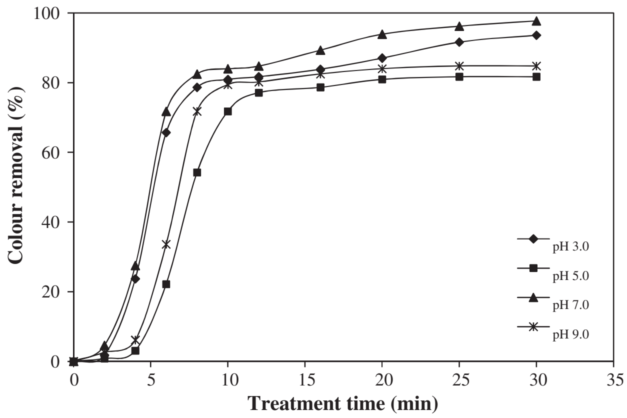

Fig. 1. Effect of pH on colour removal (Concentration of the RhB dye: 50 mg/L; Concentration of the electrolyte: 0.05 M; Current density: 15 mA/cm^2; Stirring speed: 200 rpm).

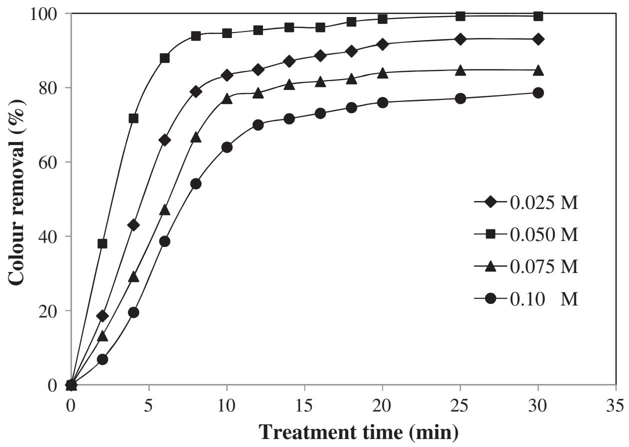

Fig. 2. Effect of supporting electrolyte concentration on colour removal (Concentration of the RhB dye: 50 mg/L; Initial pH 7.0; Current density: 15 mA/cm^2; Stirring speed: 200 rpm).

in Cl_2 and hypochlorite generation at the expense of O_2 evolution. Hence, the rate of colour removal increases. El-Ashtoukhy et al. [7] have reported that decolourization and colour removal of paper mill effluents were found to increase with the increase in NaCl concentration. An increase in the concentration of NaCl has been found to increase the generation of active chlorine [13]. In one of our earlier studies, similar results have been observed in the electrochemical oxidation of bisphenol A from aqueous solution using graphite electrodes [14]. To minimize the chloride's negative influence, an optimum concentration of 0.05 M was adopted in other experiments (Fig. 2).

3.3. Effect of applied current density on colour removal

Current density is a very important variable in electrochemical engineering. In order to investigate the influence of current density on the treatment efficiency of the electrochemical system, the different current densities from 5 to 25 mA/cm^2 were applied in this study. Fig. 3 shows that the colour removal percentage increased with electrolysis time and applied current density. In all cases, the colour removal rate was much faster during the first 20 min of electrolysis. Sharp increases of removal efficiencies were obtained with 6-min electrolysis time for current densities of 15, 20 and 25 mA/cm^2. With the current densities of 20 and 25 mA/cm^2 and at least 12-min electrolysis time, the colour removal efficiency reached higher than 90%. With the current density of 5 mA/cm^2, the process needed a longer electrolysis time to reach 90% efficiency for all electrolysis times investigated. The decolourization efficiency reached 6.88, 21.38, 59.98, 83.32 and 92.28% for the current densities of 5, 10, 15, 20 and 25 mA/cm^2, respectively, after 10-min electrolysis. After 40 min, the total removal efficiencies of colour was 93.64%, for the current density of 5 mA/cm^2, while almost complete degradation was obtained for the current densities of 10, 15, 20 and 25 mA/cm^2. It is clear that the percentage colour removal increases with increasing current density, this is attributed to the increase of Cl_2, HOCl and OCl^- concentrations in the solution, which eventually increases the dye degradation. Beyond 20 mA/cm^2, there is no significant increase in the percentage of colour removal, this may be attributed to the fact that the discharge potential Cl_2 increases with current density and becomes close to the discharge potential of O_2. Under such conditions, simultaneous evolution of O_2 along with Cl_2 takes place with a consequent reduction in the current efficiency and the rate of Cl_2 generation [17].

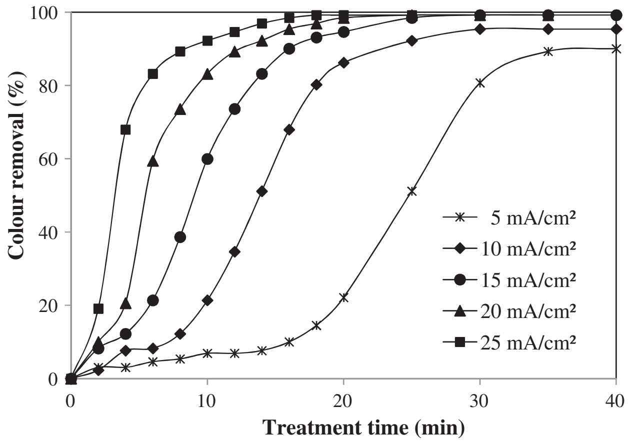

Fig. 3. Effect of applied current density on colour removal (Concentration of the RhB dye: 50 mg/L; Initial pH 7.0; Concentration of the electrolyte: 0.05 M; Stirring speed: 200 rpm).

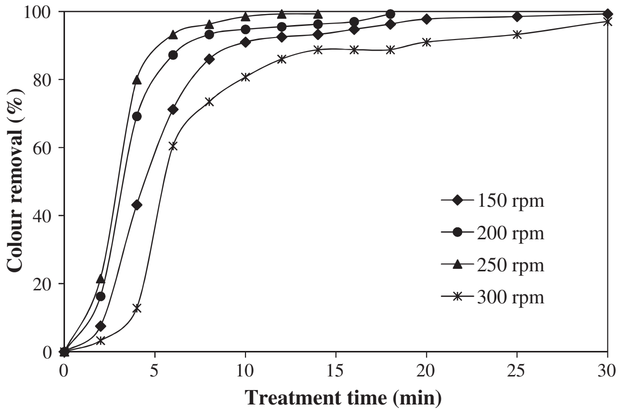

Fig. 4. Effect of stirrer speed on colour removal (Concentration of the RhB dye: 50 mg/L; Initial pH 7.0; Concentration of the electrolyte: 0.05 M; Current density: 15 mA/cm^2).

3.4. Effect of treatment time and stirrer speed on colour removal

The influence of electrolysis time was explored when the current density was kept constant at 15 mA/cm^2, supporting electrolyte concentration of 0.05 M and at initial pH 7.0. Fig. 4 shows the effect of treatment time on the colour removal. It was noted that the colour removal increased from 0 to 60 min, indicating that RhB removal was directly proportional with treatment time. The rate of degradation is high at the beginning of the process and reduces gradually to a monotonical value at the end of the process. This phenomenon was consistent with that of the electrochemical degradation of 4-chlorophenol reported by Wang et al. [18]. In view of reducing the power consumption and further optimizing the electrochemical oxidation of RhB, all other experiments were conducted with 30 min of treatment time.

In order to verify the important role of mass transfer on the electrochemical treatment of RhB solutions, the influence of stirrer speed during anodic oxidation of RhB dye solution was studied by varying the stirrer speed. Experiments were performed at four different speeds, in the range of 150–300 rpm, under the following experimental conditions; 50 mg/L of RhB dye concentration at 15 mA/cm^2 of applied current density and treatment time of 30 min. Fig. 4 shows that the percentage of colour removal decreases from 90.98 to 85.72% as the stirrer speed increases from 150 to 300 rpm. It can be seen from the results that the increasing colour removal was obtained under different stirrer speed and the colour removal is higher with the presence of stirrer. That means improving mass transfer can increase organic degradation rate. The result shows that the enhancing effect of stirrer speed on the rate of colour removal becomes less pronounced at high stirrer speeds (e.g. >100 rpm) probably because the diffusion-controlled cathodic reduction of hypochlorite and its anodic oxidation reaction are favoured at higher stirrer speed. Also, by increasing stirrer speed, colour removal is increased up to 200 rpm, beyond that there is no significance increase in colour removal. High stirrer speed leads to more rapid and high efficient electrochemical process. This confirms the fact that the colour removal reaction is diffusion controlled, the increase in rotational speed leads to the increase in the intensity of turbulence and reduces the diffusion layer thickness at the electrode surface and improves the mixing conditions in the electrolyte bulk. This enhances the rate of transfer of the reactants and products to the anode surface [19]. The electrochemical oxidation of RhB dye was found to be effective using 200 rpm as stirrer speed for other experiments.

3.5. Effect of the initial dye concentrations

The initial concentrations of dyes are one of the important factors in degradation reaction from the application point of view [20]. The variation of rate of colour removal with the initial dye concentrations is shown in Fig. 5. It can be seen from the result that the percentage colour removal decreases with the increase in initial dye concentration, this may be explained by the fact that the generated oxidizing species are identical in the same operational conditions, but they have to react with more RhB molecules and their degradation intermediates [21]. This phenomenon was consistent with some reports [14,18,22].

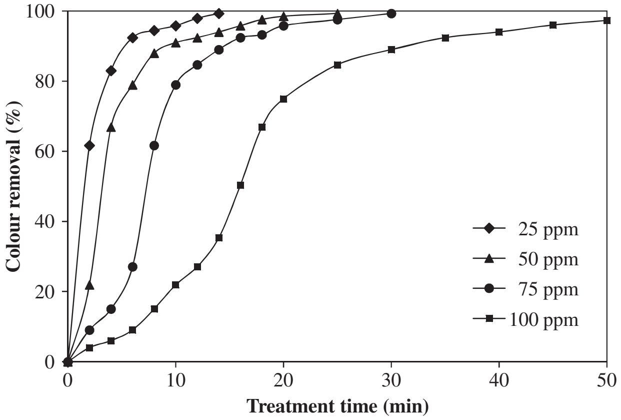

Fig. 5. Effect of initial dye concentrations on colour removal (Initial pH 7.0; Concentration of the electrolyte: 0.05 M; Current density: 15 mA/cm^2; Stirring speed: 200 rpm).

3.6. UV–vis absorbance spectra analysis

The changes in UV–vis absorbance characteristics of RhB dye during electrochemical oxidation process were investigated. During the course of experimentation, the treated samples were collected at different time intervals and the UV–vis spectra were recorded. Fig. 6 shows that a maximum absorbance peak represents the RhB dye sample and the absorbance

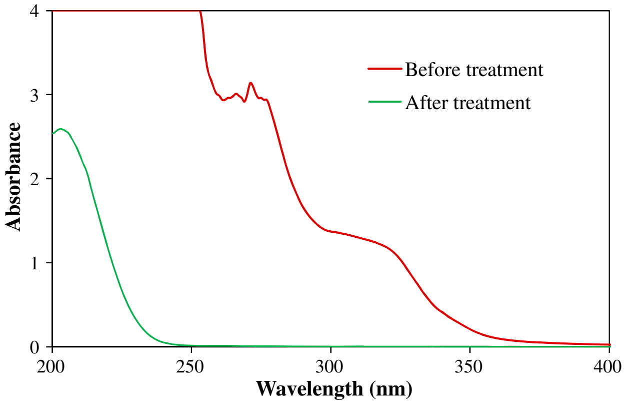

Fig. 6. UV–vis absorption spectra of RhB dye solution before and after electrochemical oxidation process using graphite electrodes (Concentration of the RhB dye: 50 mg/L; Initial pH 7.0; Concentration of the electrolyte: 0.05 M; Current density: 15 mA/cm^2; Stirring speed: 200 rpm).

gradually decreases as the treatment time of electrochemical oxidation process increases. After electrochemical oxidation process, no peak was observed at the same retention time, which confirms the removal of RhB dye from aqueous solution.

3.7. Comparison with other advanced oxidation process using graphite electrodes

Earlier studies showed that graphite has been employed for the treatment of various toxic pollutants from aqueous solution [13,14,19,22–26]. George et al. [27] studied electro-Fenton oxidation of salicylic acid from an aqueous solution. The result showed that maximum of 70% oxidation was obtained at an initial Fe^{2+} concentration of 5 mg/L, pH of 2.5, voltage of 2.5 V, flow rate of 10 mL/min and electrode spacing of 3 cm, after 10 h of electrolysis. One of our research work using graphite anodes for an electrochemical oxidation of bisphenol A from aqueous solution has achieved maximum COD removal of 78.3% under the optimized condition 120 min of electrolysis in the presence of 0.05 M NaCl with the applied current density of 12 mA/cm^2 and at initial pH 5.0 [14].

Nidheesh et al. [28] investigated the Rhodamine B (RhB) removal efficiency of graphite–graphite electro-Fenton (EF) system. The experimental results indicated that ~99% of RhB was removed after 180 min of electrolysis under the optimum pH and Fe^{2+}concentration for 10 mg/L RhB solution was found to be 3 and 10 mg/L, respectively. Nidheesh and Gandhimathi [29] studied peroxicoagulation treatment of aqueous solution containing hazardous dye, Rhodamine B, with commercially available graphite as cathode and iron as anode. The results showed that 95% of dye was removed by pH-regulated peroxicoagulation system after 180 min of electrolysis. Nidheesh et al. [30] investigated Rhodamine B (RhB) dye removal characteristics of the heterogeneous electro-Fenton (EF) process using graphite electrode. The experiment results indicated that 97.3% of RhB was removed within 180 min of electrolysis from a solution containing 10 mg/L of RhB at pH 3 with a catalyst concentration of 10 mg/L, an applied voltage of 8 V and an inner electrode spacing of 4 cm. Nidheesh and Gandhimathi [31] have conducted another research work using graphite–graphite removal of Rhodamine B (RhB) electro-Fenton (EF). The result shows that best result was achieved under the optimum experimental conditions such as applied voltage of 8 V, solution pH of 3, catalyst concentration of 10 mg/L, electrode area of 25 cm^2 and inner electrode spacing of 4 cm, 99.2% of RhB removal was obtained after 180 min of electrolysis.

4. Conclusion

Electrochemical oxidation using a graphite anode has been successfully applied to treat aqueous solutions containing Rhodamine B dye. The influence of different parameters on the removal of RhB was determined. These parameters include the pH, the current density, the supporting electrolyte concentration, stirrer speed and initial concentration of dye solution. The maximum percentage of colour removal was observed at neutral pH was beneficial for the treatment. Experimental results showed that neutral pH affects the removal of RhB and that the NaCl electrolyte concentration significantly affects the colour removal rate. According to the electrolyte concentration results, moderate (0.05 N) concentration is more suitable for dye removal with electrochemical oxidation treatment. The removal rate of colour increases with applied current density until 20 mA/cm^2 due to the increase of the mass transport caused by oxygen evolution reaction, but decreases for higher values due to the improvement of the reaction. The results indicated that the complete colour removal efficiency was achieved under the following conditions: initial pH of 7.0, current intensity of 20 mA/cm^2, supporting electrolyte concentration of 0.05 M NaCl and stirring speed of 200 rpm.

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