

Electro-oxidative Decolouration and Degradation of Amaranth Dye Wastewater in Batch Setup using Novel Ti/TiO₂-Ru₂O-IrO₂ Anode

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Abstract: In this work, electro-oxidation of amaranth dye wastewater has been performed using dimensionally stable anode (DSA) and stainless-steel cathode. Ti/TiO₂-Ru₂O-IrO₂ ternary electrode was used as the anode. Effect of various parameters such as current density, pH, voltage and time on the degradation and decolouration of dye was studied. Optimum conditions for the treatment of dye wastewater in batch setup were found. The complete decolouration was achieved in the first 5 min of the process. Decolouration and degradation of amaranth dye wastewater followed pseudo-first-order kinetics. The present research work unlocks the new horizon towards the degradation of dye wastewater employing the once through continuous process.

Key words: Amaranth, decolouration, degradation, batch setup, dimensionally stable anode (DSA), Ti/TiO₂-Ru₂O-IrO₂.

Introduction

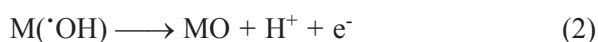
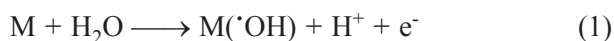
Various azo dyes are widely used as colouring agents in industries such as food, cosmetics, textile, and pharmaceuticals. The use of these chemicals makes the product highly aesthetic by imparting a beautiful colour. Regardless of that most of the dyes used for colouring purposes are proven to be highly toxic and carcinogenic in nature. Hence, there is a need to develop efficient and robust systems that can treat wastewater efficiently, continuously and effectively.

One such dye used in food, cosmetics, and pharmaceutical industries is amaranth. Amaranth is also called Acid Red 27 and imparts dark red to pink colour to the substance it is applied on depending upon

the concentration used. Dye has been classified as an endocrine disruptor and has been reported to cause an adverse effect on animal and human health (Filali-Meknassi et al., 2004; Lau et al., 2006). Endocrine disruptors are chemicals that can interfere with hormonal systems at certain doses. These disruptions can result in birth defects and cancerous tumours. The studies conducted show that amaranth possesses carcinogenic activity (Drake et al., 1975). The prolonged intake of amaranth can result in respiratory problems, birth defects, allergy and tumours (Bantle et al., 1990; Koutsogeorgopoulou et al., 1998; Lockey et al., 1959; National Research Council, 1992). Amaranth is chosen for the following study because of its versatile use and potentially harmful nature.

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From the available literature, it can be concluded that processes for the treatment of wastewater can be broadly classified as physical (Centi et al., 2002), chemical (Huang et al., 2002), biological (Cripps et al., 1990), advanced oxidation processes (AOP's) (Daneshvar et al., 2008). Physio-chemical methods such as membrane filtration, adsorption, chemical coagulation are not practical due to membrane fouling, adsorbents, and expensive chemical coagulants (Kaur et al., 2015). They also result in the generation of secondary pollutants which are usually more hazardous than a primary pollutant (Kaur et al., 2017). Among AOP's applied for wastewater treatment, electrochemical processes are most promising (Borràs et al., 2010, 2011; Boye et al., 2006; Martínez-Huitle et al., 2009). The electrochemical process is simple and requires less area compared to conventional biological treatment processes (Allen et al., 1995). Electro-oxidation was chosen over other AOP's due to its low operational cost and ease of operations in continuous setup. In recent years, the electro-oxidation processes have received tremendous attention due to their ability to eliminate a broad spectrum of organic pollutants (Martínez-Huitle et al., 2011). Electro-oxidation has the advantage of being highly versatile, efficient and eco-friendly technology and has made a huge development in its application in wastewater treatment (Sahu et al., 2015). The main advantage of the electro-oxidation process is the in-situ generation of non-selective strong oxidising species such as hydroxyl radical ($\cdot\text{OH}$). Free radical promotes the degradation of pollutants in which the rate of reaction is controlled by diffusion (Barros et al., 2014a; Hattori et al., 2003). In electrochemical treatment, both direct and indirect oxidation plays an important role. The electro-oxidation degradation of pollutants occurs either by electron transfer to the surface i.e., direct anodic oxidation or by electrogenerated ($\cdot\text{OH}$), which is formed by the discharge of water at anode surface [Equation 1] (Brillas et al., 2015). The activity of $\cdot\text{OH}$ depends upon their interaction with electrode surface M either in the form of physisorbed "active oxygen" (free $\text{M}[\cdot\text{OH}]$) or chemisorbed (oxygen in metal lattice) [Equation 2] (Anglada et al., 2009; Oturan et al., 2014).



Dimensionally stable anode (DSA) electrodes are generally made by coating electrocatalytic material containing Ti, Ru, Ir, Sn and Sb oxides (Salazar-Gastélum et al., 2013). The presence of a catalytic

coating on the surface imparts a high specific surface area to the electrodes (Salazar-Gastélum et al., 2013). This electrode shows excellent mechanical and chemical resistance at high current densities and strongly acidic media (Salazar-Gastélum et al., 2013). Song and Xiong (2007) stated that DSA such as $\text{PbO}_2\text{-CeO}_2$ can work effectively for several years under normal conditions. DSA electrodes were used for the treatment of herbicides (Neto et al., 2009). The use of these electrodes for the treatment of highly toxic chemicals such as cyanides was reported (Lanza et al., 2002). The application also involves its use in treating dye wastewater (Rajkumar et al., 2006). The nature of oxidising species generated depends upon the type of coating applied on the surface of the electrode. DSA is classified as binary such as Sn-S, Ir-Ru, Ir-Ta, Ti-Ru, and Ti-Bi electrodes, tertiary such as Ir-Ru-Ti, Ce-Ru-Sn, and Ir-Ru-Sn and quaternary such as Ru-Ir-Sn-Ti electrode (Wu et al., 2014).

From the literature survey, it has been observed that research groups have presented the use of DSA such as SS/ PbO_2 (Elaissaoui et al., 2019), Ti/Sn-Ir-Ru (Oturán et al., 2014), Ti/Ru-Sn (Oturán et al., 2014), Ti/Pt (Fajardo et al., 2017), Ti/Pt-Sn-Sb (Dbira et al., 2016) for the degradation of amaranth dye wastewater. The use of materials such as the PbO_2 film as the anodic coating can cause a potential hazard to the environment due to the chances of peeling (Singla et al., 2017). Ti/ $\text{TiO}_2\text{-Ru}_2\text{O}_3\text{-IrO}_2$ ternary DSA novel electrode has been used in the present study, which is a combination of the Ti/ $\text{TiO}_2\text{-RuO}_2\text{-IrO}_2$ ternary electrode and has good resistance properties of Ir-Ru. The inclusion of Ir-Ru in electrode makes the surface uniform, smooth with minor cracks, which helps in preventing corrosion (Gupta et al., 2012).

Decolouration of dye does not necessarily result in the degradation of dye. It can be present in the form of intermediates which are often more hazardous than dye itself, thus it is necessary to analyse decolouration as well as degradation of dye at peaks in the visible as well the UV region. In most of the studies reported in the literature to treat the amaranth dye wastewater using the electrochemical method, only decolouration was analysed. In the spectrum for calibration of dye, the peak lying in the visible region signifies the visible colour of dye and the peak lying in the UV region signifies the chemical structure of the dye. The peak at 521nm indicates -N=N- chromophore (Barros et al., 2014a,b; Oturan et al., 2014). The peak at 243.50 nm, which falls within the infrared region and represent $\pi\text{-}\pi^*$ transitions in aromatic rings conjugated with azo bonds (Barros et al., 2014a,b). So, the decolouration

and degradation were analysed at 521 nm and 243.50 nm, respectively.

To the best of our knowledge, this is the first reported study where the once through process has been used for the degradation of amaranth dye using dimensionally stable $\text{Ti/TiO}_2\text{-RuO}_2\text{-IrO}_2$ ternary electrode. Once through continuous systems have more potential for industrial applications. In the present study, analysis is conducted on both batches and once through a continuous system.

Experimental

Reagents and Chemicals

All chemicals used in the experiment were of analytical grade. Amaranth or Acid Red 27 dye (Sigma Aldrich, Mexico) of analytical grade was used for this study. NaCl (MERCK Ltd, India) was used to maintain the conductivity of the solution. To balance the pH of the solution, HCl and NaOH (MERCK Ltd, India) was used. All experiments were carried out using double-filtered deionised water (Jyoti Enterprises, Jaipur, India).

Experimental Setup and Procedure

D.C. power supply (0-35 V, 0-10 A) was used for providing potential difference (purchased from Testronics, Pune, India). DSA i.e. the $\text{Ti/TiO}_2\text{-Ru}_2\text{O-IrO}_2$ electrode (purchased from Exotic Elements Pvt. Ltd. Mumbai, India) of dimension 70 mm×70 mm×1

mm was used. Pure stainless steel of SS grade 304 was used as an anode as it is inactive in the main reaction. A constant stirring rate of 120 rpm was maintained in all experiments using a magnetic stirrer (purchased from Remi, India) on the batch as well as a continuous setup. Analysis of samples collected during the process as well as spectrum analysis was conducted by using a spectrophotometer (purchased from Shimadzu, Japan). For a continuous experiment setup, a peristaltic pump (0 to 99 ml min^{-1}) was used (purchased from Ravel Hiteks, Chennai, India). Wastewater dye having a dye concentration of 50 ppm and the electrolyte concentration of 0.08 mol L^{-1} were used for both batch and continuous setup experiments.

The experimental setup used for batch experiments has been shown in Figure 1. A 500 mL beaker was modified to suspend and hold electrodes in place during the experiment. The batch reactor was placed on a magnetic stirrer. The gap between the electrodes was maintained by using non-conducting, concentric PTFE tubes. The electrical current was provided by attaching the wires to the DC power supply. Samples were drawn from the same spot during each experiment.

For accurate detection of the concentration of dye in the sample, a relationship between concentration and absorbance was established at peak wavelengths of 521 and 243.50 nm. Figure 2 shows the UV spectrum of amaranth dye. At 521 nm, the linear equation of

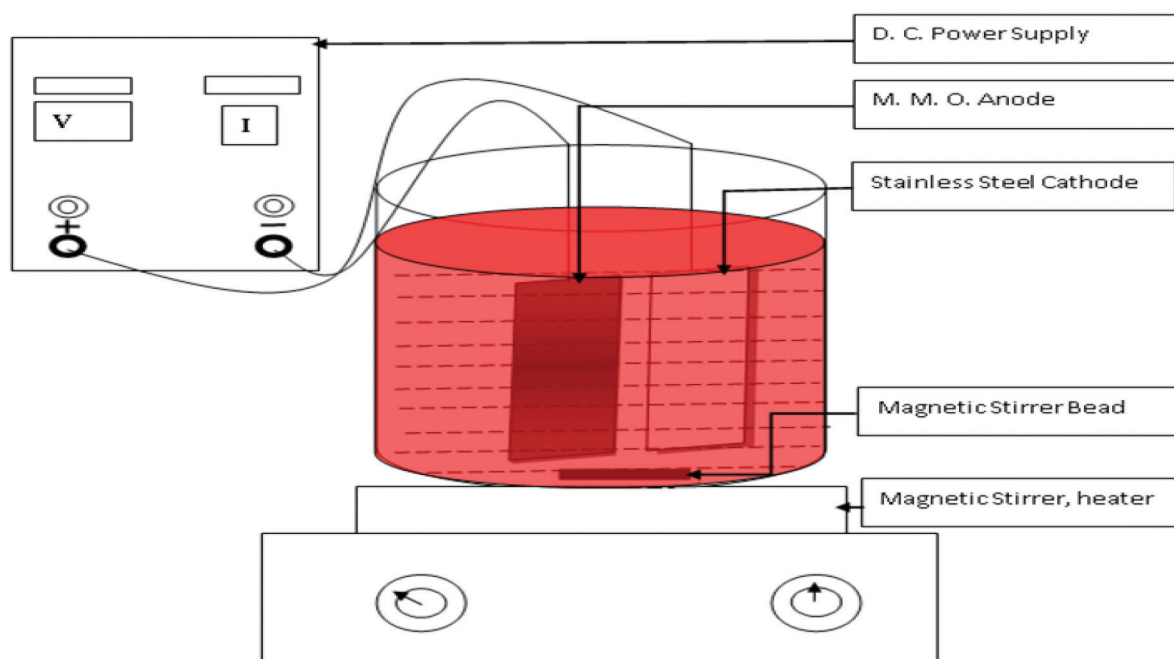


Figure 1: Schematic representation of batch setup.

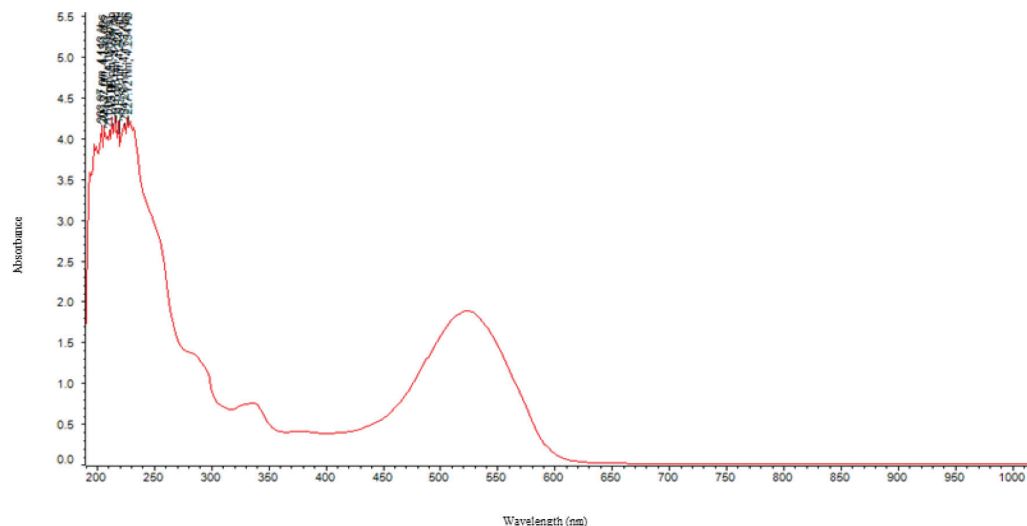


Figure 2: UV spectrum of amaranth dye.

regression is concentration = absorbance/0.0396 with the R^2 value of 0.9972. At 243.50 nm, the linear equation of regression is concentration = absorbance/0.0548 with the R^2 value of 0.9945.

The percentage decolouration (% decolouration) was calculated at 521 nm as follows:

$$\% \text{ Decolouration} = 1 - \frac{\text{final conc. at 521 nm}}{\text{initial conc}} \times 100 \quad (3)$$

The percentage of dye degradation (% degradation) was calculated as follows:

$$\% \text{ Degradation} = 1 - \frac{\text{final conc. at 243.50 nm}}{\text{initial conc}} \times 100 \quad (4)$$

Energy consumption (E.C.) in kWh kg⁻¹ dye was calculated by the formula

$$\text{E.C.} = \left[\frac{V \cdot I \cdot t \cdot 1000}{60 \times \text{mg.dye degraded at 521 nm}} \right] \times 100 \quad (5)$$

Where V = applied voltage, I = applied current and t = time of treatment in min and E.C. = energy consumption in kWh kg⁻¹ dye degraded.

The mg dye degraded was found from the dye concentration at 521 nm.

Results and Discussion

Electrochemical Decolouration and Degradation of Amaranth

The pH of the solution is one of the most important factors that affect the performance of the electro-

oxidation process. It describes the type of Cl^- species which is predominant and also the rate of absorption of $\cdot\text{OH}$ (Kumar et al., 2007). The effect of pH on the decolouration of dye was studied. The experiments were conducted under acidic, initial, basic conditions of pH 3, 6.30 and 9, respectively. Effect pH on the decolouration and degradation of amaranth dye wastewater is as shown in Figure 3(a,b).

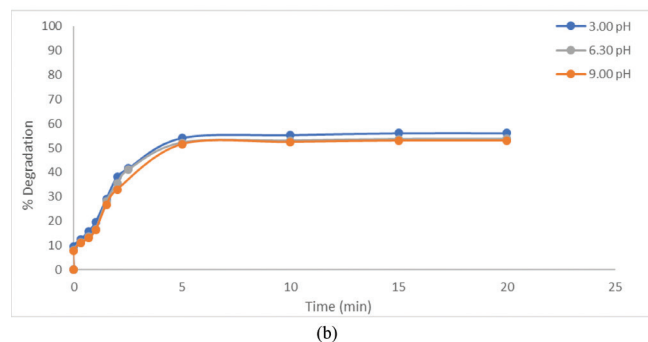
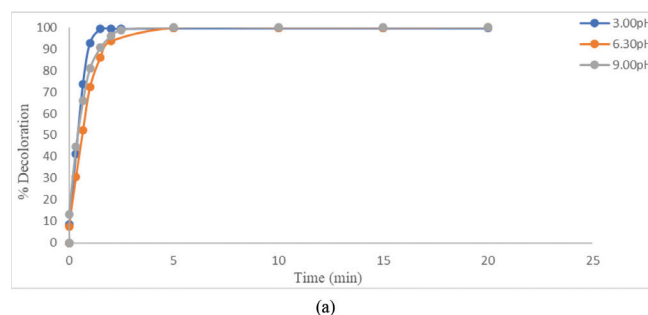


Figure 3: Effect of pH decolouration and degradation of amaranth dye wastewater. (a) % decolouration vs. time and (b) % degradation vs. time.

From the results, it was found that the % decolouration decreases with increasing the pH; however, the effect of increasing the pH is small. From Figure 3b, it can be seen that the % degradation increases with decrease pH but the effect of pH on degradation is small. Earlier studies reported that degradation increases with a decrease in pH (Gupta et al., 2012). At lower pH, the adsorption rate of $\cdot\text{OH}$ is high which oxidises the pollutant through mediated oxidation (Singla et al., 2018). On the other hand, at a basic pH, $\cdot\text{OH}$ is converted to H_2O_2 and HO_2 radicals, which are oxidants of lower potential and result in a decreasing rate of oxidation (Singla et al., 2017). As indicated in Figure 3 (a,b), pH has a little effect on decolouration and degradation, hence, it was decided to carry on further analysis on initial pH only.

Voltage is a crucial factor in any electrochemical process as it affects energy consumption. Energy consumption further affects the viability and applicability of the process in real life. Hence, it is important to study the effect of applied voltage on the process. To determine the effect of voltage on the process, the voltage was varied using the DC power supply. The experiments were carried out on five different voltages of 2.5 V, 3 V, 3.5 V, 4 V, and 4.5 V, respectively.

From Figure 4a, it can be seen that as the voltage increases, the % decolouration increases till the voltage of 3.5 V is attained, then it further increases beyond 3.5 V to 4 V and 4.5 V, thereby producing little effect on the process. Figure 4b shows the effect of voltage on % degradation. It can be seen that as the voltage increases so does the degradation. A similar observation on the effect of voltage on the decolouration of amaranth has been made by Salazar-Gastélum et al. (2013).

An increase in voltage increases % degradation; however, the cost of the process also increases. As shown in Figure 4a, an increase in the voltage beyond 3.5 V has a marginal effect on decolouration, thus, a measure of 3.5 V was taken as an optimum condition for all further experiments.

Current density is another important operating parameter considered for the electrochemical process. It affects the cost of the process as well as the mechanism of the reaction. The rate of generation of oxidative species such as $\cdot\text{OH}$, HO_2 and also chloro-oxidative species such as Cl_2 , ClO^- , ClO_3^- , and ClO_4^- was considered for this study. Dbira et al. (2016) found that the rate of generation of chloro-oxidative species significantly increased with an increase in current density. To exactly study the effect of current density on decolouration and degradation, optimum and constant

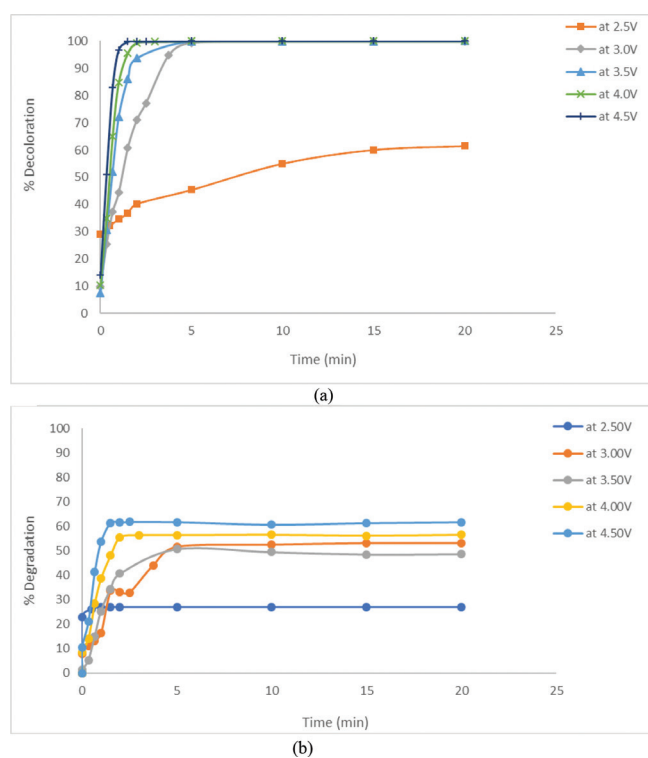


Figure 4: Effect of voltage on decolouration and degradation of amaranth. (a) % decolouration vs. time and (b) % degradation vs. time.

voltage of 3.5V was selected and experiments were carried out at current densities ranging from 7.55 to 3.47 mA cm^{-2} . Figure 5a shows the effect of current density on decolouration. It can be seen that on increasing the current densities, the rate of decolouration increases. Figure 5b shows the effect of current density on the % degradation. It can be seen for the rate of degradation, decolouration increases with an increase in current density; similar observations are made by Steter et al. (2014). It was observed that the % degradation decreases with an increase in current density. An increase in current density may lead to the occurrence of side reactions that consumes $\cdot\text{OH}$, thus decreasing the % degradation in the process (Steter et al., 2014). This indicates that degradation and decolouration are two different processes.

Based on the energy consumption, 6.53 mA cm^{-2} was taken as an optimum condition for further experiments.

To investigate simultaneous decolouration and degradation of dye with time, the experiment was conducted under the optimum conditions of 3.5 V and 6.53 mA cm^{-2} . Figure 6 demonstrates the complete spectrum of treated samples at different time intervals.

Complete decolouration of amaranth was achieved within 3 mins of treatment; this was faster as compared to the previous attempts made to treat the amaranth

dye using electrochemical methods. Salazar-Gastélum et al. (2013) reported that the complete decolouration took place in 5 min. Barros et al. (2014b) reported the complete decolouration in 90 min. Steter et al. (2014) reported 99% decolouration by combining electrochemical and ultrasonic cavitation in 90 min.

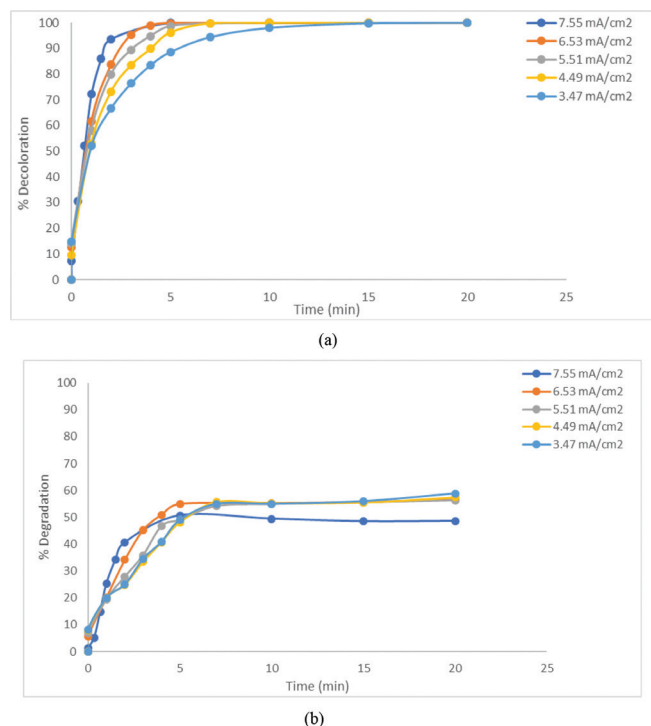


Figure 5: Effect of current density on decolouration and degradation of amaranth dye. (a) % decolouration vs. time and (b) % dye degradation vs. time.

Elaissaoui et al. (2019) reported complete decolouration in 120 min. Barros et al. (2014a) studied different conditions for the electro-Fenton degradation of food dye amaranth using a gas diffusion electrode modified with cobalt (II) phthalocyanine in which highest decolouration was reported using 0.15 mM Fe²⁺ and it was 95% at 90 min.

It was found that the degradation of the dye i.e. its decay at 243.50 nm was 60% at 2.5 min. The slow degradation of the chemical structure can be attributed to the slow degradation of dye intermediates formed in process (Elaissaoui et al., 2019).

Energy Consumption

Energy consumption is a parameter that determines the economic and operational viability of the process. To make the process energy efficient it is necessary to minimise the energy consumption. Energy consumption directly affects the operational cost of the process. To minimise energy consumption, it is necessary to operate the process under optimum conditions of voltage and current. In our case, the optimum conditions such as potential difference and current densities are 3.5 V and 6.53 mA cm⁻², respectively. Energy consumption is calculated in kWh kg⁻¹ of dye by using Equation 5.

Energy consumption for 99.9 % removal of dye is 1.51 kWh kg⁻¹, which is much less compared to the previous attempts made to degrade the dye using the electrochemical method. Salazar-Gastélum et al. (2013) reported energy consumption was 5 kWh kg⁻¹ for complete decolouration of the dye.

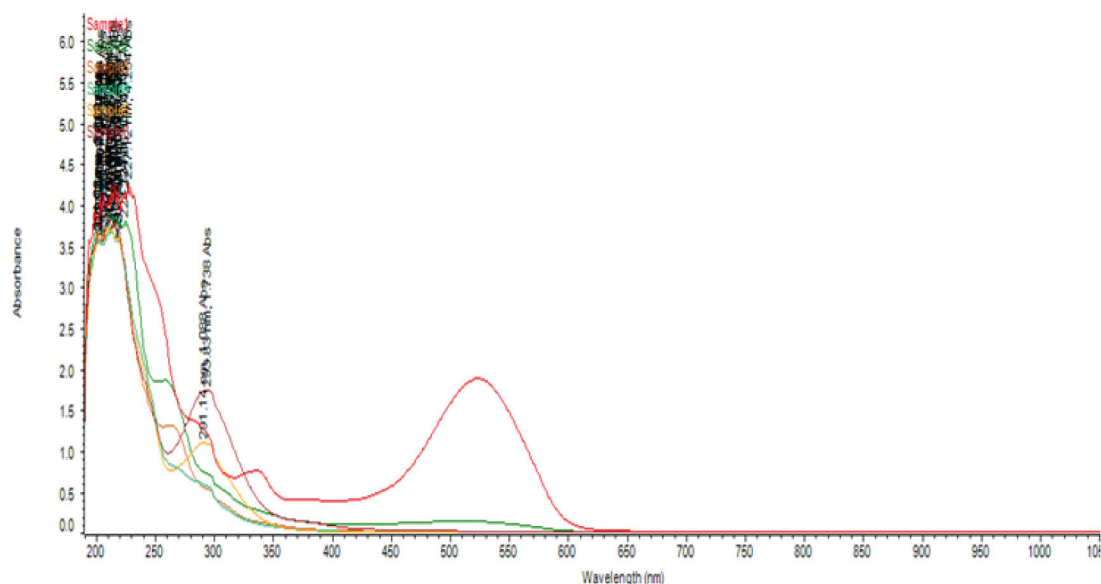


Figure 6: Spectral analysis of treated samples collected at 0, 2, 5, 10, 15 and 30 min.

Kinetics of Reaction

Kinetics of Reaction in Batch Setup

The kinetic study of reaction gives information about the rate and order of the reaction. Kinetic study were performed for decolouration and degradation of dye under optimum conditions. The data were fitted according to pseudo-first-order kinetics. The rate equation for pseudo-first-order kinetics is as follows:

$$-\frac{dC}{dt} = kC \quad (6)$$

On integrating, we get,

$$-\ln \frac{C_t}{C_o} = kt \quad (7)$$

The graph between $-\ln(C_t/C_o)$ and time (t) is plotted. Figure 7a shows the graph between $-\ln(C_t/C_o)$ and time for decolouration, while Figure 7b shows the graph between $-\ln(C_t/C_o)$ and time for degradation. For decolouration, the k value is 1.2746 min^{-1} and the R^2 value is 0.9889, which indicates a near-perfect fit. For degradation, the k value is 0.2737 min^{-1} and the R^2 value is 0.982. This indicated that decolouration and degradation both follow pseudo-first-order kinetics. The results are in conjunction with observations made by Barros et al. (2014b); Elaissoui et al. (2019); Salazar-Gastélum et al. (2013); Steter et al. (2014). The value

of k was obtained as 1.2746 min^{-1} for decolouration and 0.2737 min^{-1} for degradation. This indicates that the process of decolouration is faster than degradation.

Conclusion

Degradation and decolouration of amaranth dye wastewater in batch and once through the continuous system using titanium-based DSA has been studied successfully. Optimum values of pH, voltage, current density, and flow rate were found to be 6.3, 3.5 V, 6.53 mA cm^{-2} and 60 ml min^{-1} , respectively. In the batch process, complete decolouration was achieved in the first 3 and 5 minutes of the process respectively. Due to the slow degradation of intermediates, the maximum degradation achieved was 65% in the process. The study conducted showed that amaranth dye wastewater can be treated by using the $\text{Ti/TiO}_2\text{RuO}_2\text{IrO}_2$ anode. Electrical consumption for complete decolouration of the dye was found to be 1.8 kWh kg^{-1} at optimum values of voltage and current density. Kinetics of decolouration and degradation were studied in batch processes and it was found that the process follows pseudo-first-order kinetics. A study conducted showed that electro-oxidation can be used successfully to treat amaranth dye wastewater.

Acknowledgement

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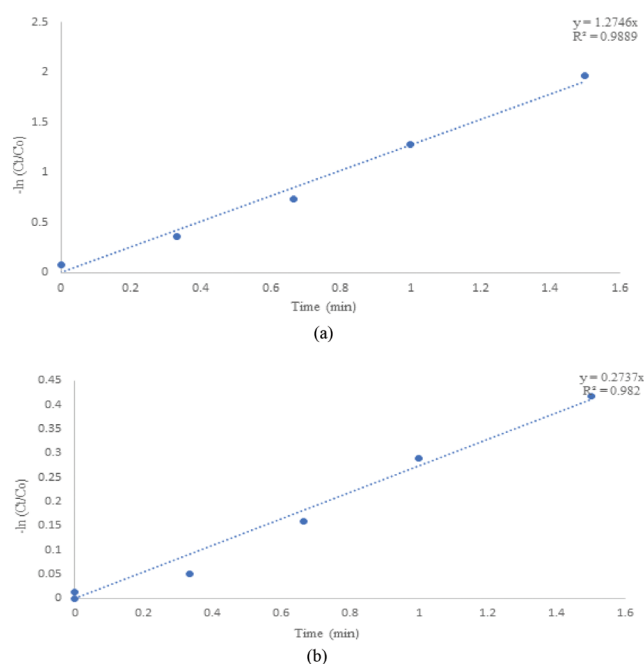


Figure 7: Kinetics of reaction in batch setup (a) $-\ln(C_t/C_o)$ at 521nm vs. time and (b) $-\ln(C_t/C_o)$ at 243.50 vs. time.

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