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Electrochemical degradation of aqueous solution of Amaranth azo dye on ACF under potentiostatic model

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Abstract

The Amaranth azo dye degradation with electrochemical method in aqueous solution has been studied under potentiostatic condition with activated carbon fiber (ACF) electrode. Cyclic voltammetry on platinum (Pt) wire and quasi-steady-state polarized curves on ACF held by a Pt wire were investigated at first. The mechanisms of the electrochemical processes for Amaranth on ACF were defined as adsorption, electrooxidation and electroreduction based on the changes in the ratios of normalized absorbency values. The electrooxidation potential and electroreduction potential were, respectively, ascertained at 600 mV and −200 mV. The adsorption of the dye on ACF was found insignificant. Successes in removal of the color, chemical oxygen demand (COD) and total organic carbon (TOC) were demonstrated when a proper potential region was selected. The relationship between the decolorization and applied potential was obtained. For electroreduction, it was found that the dye with the initial concentration of 80 mg L^{-1} was able to be completely removed after 5 h at the potential of −600 mV. At the same time, COD and TOC contents were both reduced by about 60%. For electrooxidation, the color removal was 95.4% at the potential of 1000 mV with the COD and TOC removal ratios being about 35% and 30%. The spectra of the dye were analyzed before and after treatment and confirmed that the azo bond −N=N− was destroyed in the range of −800 mV to −200 mV and 600 mV−1400 mV.

Keywords: Potentiostatic model; Activated carbon fiber (ACF); Dye; Decolorization; Electrooxidation; Electroreduction

1. Introduction

Wastewaters originated from textile industry contain various pollutants including a high content of organic matter and suspended masses and dissolved salts [1]. If improperly processed, these effluents not only deteriorate the aesthetics of waters and hinder the penetration of oxygen, but also pose significant threat to life forms upon hydrolysis of some dyes in the wastewater to form toxic products [2]. In some cases less than 1 mg L^{-1} of dye concentration produces an obvious water colorization [3]. Dyestuffs are highly structured polymers with low biodegradability [4]. Among the dyes' family, more than half are azo compounds. Hence, a lot of research has been focused on methods of azo dye destruction.

There are many treatment processes that have been used or investigated extensively to treat textile wastewaters such as biological treatment [5], chemical precipitation [6], adsorption by activated carbon [7], photocatalytic oxidation [8], ozonation [9], Fenton's reaction [10] and electrochemical methods [11,12].

Electrochemical method is simple. The process requires significantly less area than conventional biological treatment processes [13]. For electrochemical treatment of dyeing wastewater, direct oxidation and indirect oxidation processes both play an important role. Naumczyk et al. [14] investigated the process of electrochemical treatment of wastewater containing

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dyes in the presence of high concentration of chlorides. They proved that Ti/RuO_2, Ti/Pt, and Ti/Pt/Ir electrodes were very effective in direct or indirect oxidation of textile wastewater. Yang et al. [15] investigated the hypochlorite generation on Ru–Pt binary oxide for dyeing wastewater treatment. Jüttner et al. [16] suggested that in the presence of chloride the oxidation for azo dyes happened mainly through the formation of 'active' chlorine. Vlyssides et al. [17] investigated Ti/Pt as anode and stainless steel 304 as cathode in wastewater treatment of reactive azo dyeing process. Chen et al. [18] studied Orange II and 16 reactive dye mineralization with Ti/B–diamond and found that the electrode was much more active than Ti/Sb_2O_5–SnO_2. Galvanostatic model was applied in all the works above for electrochemical process research and the method by controlling constant current density was used widely since current is the key factor for reaction rate.

In several works, d.c. power source was used and the purpose was to provide a cell voltage and to investigate the effect of the final treatment for the application used such as by Öğütvren et al. [19] and Fan et al. [20]. Generally speaking, potentiostatic model was not used as widely as galvanostatic model and cell voltage control. Cerón-Rivera et al. [21] tested four different electrode materials: diamond–, aluminium–, copper– and iron–zinc alloy electrodes by applying different potentials in the range −1.0 V to −2.5 V with Basic Yellow 28 and Reactive Black 5 being model dyes. Dávila-Jiménez et al. [22] used Pt, Ti and diamond as working electrodes for dyeing water treatment and found that electrochemical treatment using a diamond electrode is an effective method for the study of decomposition of textile dyes of different nature. Three potentials of 2.2 V, 2.5 V and −2.5 V in the cited order were applied. However, limited potentials were tested and no enough information about the decolorization and potentials were provided.

Activated carbon fiber (ACF) is used as a type of three-dimensional electrode. As a result, the energy efficiency of reaction can be greatly enhanced. The particularly high specific areas obtainable with ACF makes it an ideal electrode for removal of pollutants, especially organic molecules from effluents. At the same time, there are many functional groups on its surface, such as C=O that makes ACF an effective adsorbent for pollutants. The adsorbed species can react with the functional groups physically and chemically when polarized. The degradation of more than 25 dyes for decolorization including azo and anthraquinone dyes was studied [23,24]. ACF + Fe and ACF were used as anode and cathode, respectively. Almost all dye solutions tested were decolorized effectively in the electrochemical system. Because only a single-compartment cell was used, it is difficult to judge the role of each electrode in the reaction. Hence no specific conclusion was obtained for the treatment on ACF or Fe. In addition, d.c. power source was used to provide cell voltage and the important parameter of potential which is the motive force for electrolysis was not considered. Since Fe electrode can release Fe^{2+} when a voltage was given, electrocoagulation may contribute to the color, TOC and COD removal [25,26]. It is therefore necessary to carry out other research on the effect of ACF used as working electrode without the coagulation effect.

The objective of this work is to investigate the effect of potential on the azo dye decolorization. Constant potential was controlled to evaluate the effect of potential on dye solution degradation. The experiment was conducted in a cell separated by a cation selective membrane. The electrochemical process was conducted under positive and negative potentials. Pseudo-first order degradation kinetics was obtained. The empirical relationship between the rate constant and the potential was provided in the negative potential range. At the same time, chemical oxygen demand (COD) and total organic carbon (TOC) removal was tested. Moreover, the spectra of the dye effluents before and after treatment were analyzed for the information about dye structural destruction.

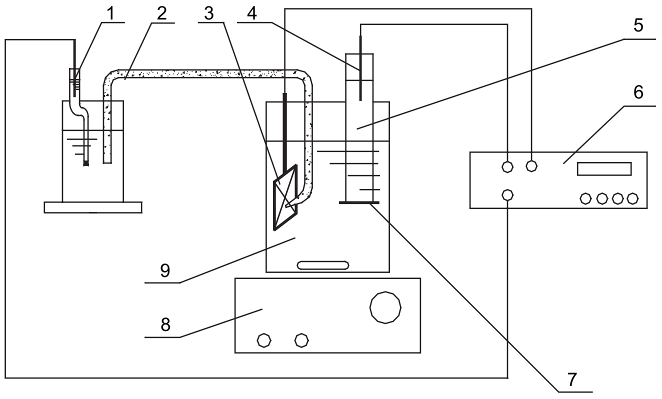
2. Materials and methods

2.1. Experimental facility

The experimental set-up employed in this study was a conventional three-electrode cell shown in Fig. 1. The working electrode was a weighted piece of ACF held by a Pt wire. The counter electrode was a Pt wire. A saturated calomel electrode (SCE) was used as the reference electrode. The cell was divided into two compartments with a cation selective membrane. The solution was stirred by a magnetic bar. A potentiostat/galvanostat (Shanghai REX Instrument Factory, China) was used in controlling the potential. The UV–vis spectra were analyzed with a UV–vis Spectrophotometer (Jasco V-560, MHT-344, Japan). TOC was estimated with the Shimadzu model TOC-V_{CPH} analyzer (Japan). COD content of all samples was determined according to standard method for the examination of water and wastewater [27].

2.2. Materials

Amaranth was used as the model compound whose structure and spectrum are shown in Fig. 2. This azo dye was



1. Reference electrode, 2. Salt bridge, 3. Working electrode (ACF held by Pt wire), 4. Counter electrode (Pt wire), 5. Electrochemical auxiliary room, 6. Potentiostat, 7. Cation selective membrane, 8. Magnetic stirrer, 9. Electrochemical working room.

Fig. 1. Schematic diagram of the electrochemical set-up.

purchased from Huizhou Fond Food Additives Co. Ltd., Guangdong, China and used as received. All other chemicals used were of analytical grade. Na_2SO_4 ($0.1\ mol\ L^{-1}$) was used as the supporting electrolyte; $3.53\ g\ L^{-1}$ HPO_4^{2-} and $3.39\ g\ L^{-1}$ $H_2PO_4^-$ were used to adjust the pH to about 6.6. As can be seen from Fig. 2 the maximum absorption for the dye in the visible region was at 521 nm and there is also characteristic absorption peak at the wavelengths of 280 nm and 330 nm in the ultraviolet region.

The ACF used in this study was provided by Anshan Activated Carbon Fiber Company, Liaoning, China, having a specific area of $1180\ m^2\ g^{-1}$. Pt wire (≥99.95% in purity) was provided by Shanghai Chemical Reagent Company, China National Pharmaceutical Group.

The ACF was first washed with double boiled deionized water. Then it was dried under vacuum at 120 °C for 2 h and cooled in a desiccator. The ACF was cut in desired dimensions, weighed accurately and attached to the Pt wire before dipping into the reaction cell.

2.3. Experimental conditions

The initial solution was $80\ mg\ L^{-1}$. The processes of electrochemical polarization were examined under potentiostatic model. ACF was used as anode and cathode. The range of potential applied was from −800 mV to 1400 mV. Each test lasted for 8 h with 1 mL sample taken out every 1 h for absorbency value analysis. The COD, TOC and UV–vis spectra of the samples were analyzed after each batch of test. Temperature was kept at 20 °C and the solution was stirred with the same speed of 240 rpm.

3. Results and discussion

Color, COD and TOC removal ratios (%) were calculated as follows

$$X_{\text{color}} = \frac{A_0 - A_t}{A_0} \times 100\% \tag{1}$$

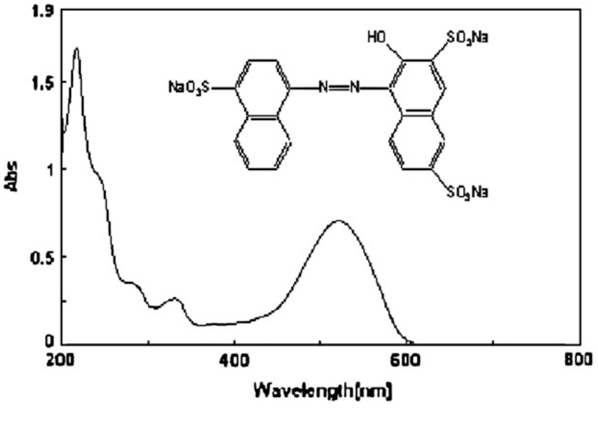


Fig. 2. UV–vis spectrum of initial Amaranth solution and molecular structure of Amaranth in non-hydrolyzed form (initial solution concentration: $80\ mg\ L^{-1}$; dilution ratio = 3:1; Maximum wavelength: 520 nm, 330 nm, 280 nm; Amaranth: Index number: C. I. Food Red 9 (16185)).

where A_t and A_0 are the absorbency values (Abs) at 520 nm at time t and 0, respectively.

$$X_{\text{COD}} = \frac{\text{COD}_0 - \text{COD}_\text{t}}{\text{COD}_0} \times 100\% \tag{2}$$

where COD_0 and COD_t are the COD values before and after treatment, respectively.

$$X_{\text{TOC}} = \frac{\text{TOC}_0 - \text{TOC}_\text{t}}{\text{TOC}_0} \times 100\% \tag{3}$$

where TOC_0 and TOC_t are the TOC values before and after treatment, respectively.

The ratios of normalized absorbency values were calculated with Eq. (4)

$$Z(521/X) = \frac{A'_{521}}{A_{521}} \bigg/ \frac{A'_x}{A_x} \tag{4}$$

Where $Z(521/X)$ – ratio of normalized absorbency values; x – 280 nm or 330 nm, A_{521}, A_x – absorbency values of initial dye solution; A'_{521}, A'_x – absorbency values of dye solution after treatment.

3.1. Adsorption effect

In order to estimate the contribution of pure adsorption, open-circuit experiments were investigated first. The results show that there is some adsorption but the capacity is about $17\ mg\ g^{-1}$ ACF after 8 h at an initial condition of $80\ mg\ L^{-1}$. The experiment in the range of −100 mV to 500 mV was also conducted. The results show that in such potential range, the adsorption capacity has no obvious increase by comparison with the value under open-circuit condition. The load of ACF was fluctuated by around $25\ mg\ g^{-1}$ ACF. The polarization has little effect on the abatement of color. The adsorption and electrosorption [28,29] were insignificant for Amaranth.

The mechanisms of the decolorization under different potentials for the dye were estimated based on the ratios of the normalized absorbency values. If the ratios of the normalized absorbency values were equal to 1.0, then the dye structure was kept intact. Otherwise, the dye structure was destroyed and the electrochemical reaction happened [30]. Table 1 shows the results of the ratios of the normalized absorbency values in all potentials controlled and a conclusion can be made that in the range of −100 mV to 500 mV, the ratios were equal to 1.0 and no chemical reaction took place. Just in this region, the decolorization results from electrosorption. In the range of 600 mV–1400 mV, the ratios were lower than 1.0 and the decolorization resulted by electrooxidation. Similarly, in the region of −200 mV to −800 mV, the ratios changed a lot and the decolorization resulted from electroreduction.

Table 1
The results of normalized absorbency values for Amaranth during the process of electrochemical decolorization under potentiostatic model

Potential *V*	Normalized absorbency *Z*(521/280)	Normalized absorbency *Z*(521/330)	Potential *V*	Normalized absorbency *Z*(521/280)	Normalized absorbency *Z*(521/330)
Original	1.0	1.0			
Open circuit	1.0	1.0	0.3	1.0	1.0
−0.8	*0.01*	*0.01*	0.4	1.0	1.1
−0.7	*0.01*	*0.01*	0.5	1.0	1.0
−0.6	*0.05*	*0.06*	***0.6***	***0.64***	***0.70***
−0.5	*0.04*	*0.05*	*0.7*	*0.19*	*0.24*
−0.4	*0.36*	*0.46*	*0.8*	*0.06*	*0.07*
−0.35	*0.83*	*0.92*	*0.9*	*0.18*	*0.22*
−0.3	*0.81*	*0.86*	*1.0*	*0.08*	*0.11*
−0.2	***0.85***	***0.91***	*1.1*	*0.09*	*0.12*
−0.1	1.0	1.0	*1.2*	*0.17*	*0.20*
0.1	0.93	1.0	*1.3*	*0.15*	*0.18*
0.2	1.0	1.0	*1.4*	*0.22*	*0.28*

3.2. Electrochemical behavior of Amaranth on Pt wire and ACF electrode

The cyclic voltammetry (CV) of the dye on Pt wire was done from −1100 mV to 400 mV at a scanning rate of 25 mV s^{-1} (Fig. 3). The CV curves of Amaranth and blank solution are quite similar with no peaks observed during the scan at positive potential. For the dye solution at about −800 mV, however, there is a peak that begins at −600 mV. The peak observed at about −900 mV signifies the start of hydrogen evolution. Hence, −600 mV was the hydrogen atom adsorption potential and −800 mV was its maximum. These two values are very important in dye removal as will be discussed subsequently.

Fig. 4 shows the polarized curves of Amaranth and blank solution at quasi-steady-state on Pt wire and ACF held by Pt wire. For the positive polarization, it is expected that the Pt wire in the blank or dye solution do not show any difference because Pt wire has little effect on the reaction in the positive potential range and only to provide even potential for the ACF. These results are consistent with the CV of the dye on Pt wire above. It is interesting to note that in blank solution the current on ACF started to increase since 600 mV and almost kept at the platform of 1700 mV. When dye is used, the increase of current started at the same potential and an anodic peak at about 1000 mV can be seen clearly with the current of 4.5 mA. Oxygen evolution is responsible for the steep rise in current at about 1500 mV. In the region of 600 mV–1400 mV, some surface changes may take place to make electrochemical degradation as seen in the subsequent section. For the negative polarization, there is no peak and almost no difference for ACF in blank or dye solution.

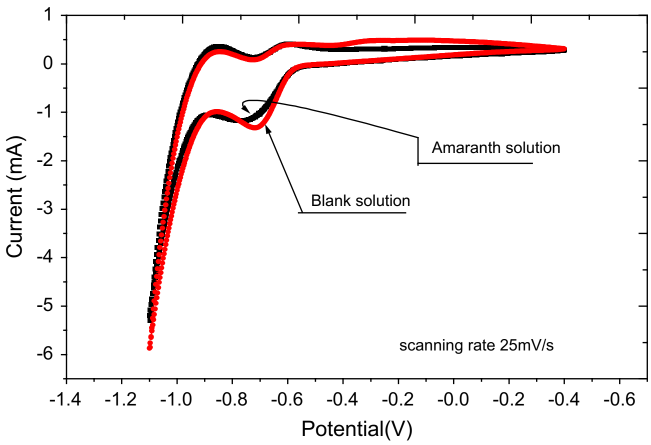


Fig. 3. Cyclic voltammograms of Amaranth on Pt wire electrode.

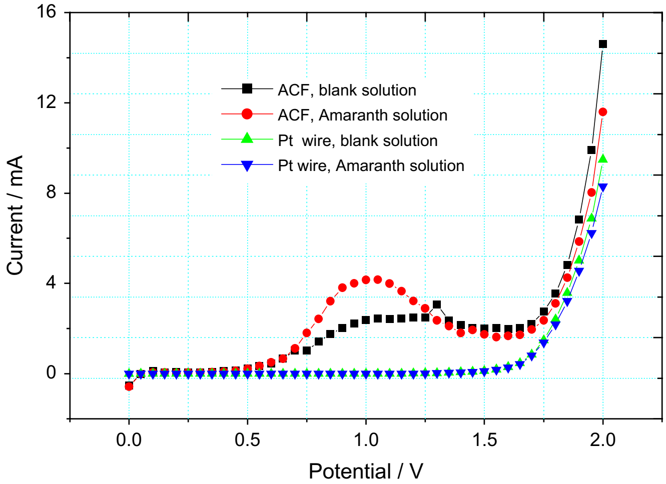


Fig. 4. Quasi-steady-state polarized positively curves for Amaranth on Pt and ACF electrodes.

By analyzing the curves above, the dye was probably oxidized since 600 mV on ACF. However, there were no reductive peaks on ACF.

3.3. Process of electrooxidation

3.3.1. Kinetics of the electrochemical process

The result of the color removal on time for electrooxidation is shown in Fig. 5. The final color removal ratio at 600 mV is about 42.7% which is over 7 times of that at open circuit. If the potential was up to 1000 mV, the color was decreased 95.4%, which was about 17 times of that at open circuit of 5.7%. Further increase in the potential leads to decrease in the color removal to 88.8% and 84.0% at 1200 mV and 1400 mV, respectively. The result is consistent with the quasi-steady-state curves on ACF for the Amaranth. It was supposed that the functional group on ACF underwent some reactions when polarized at a certain potential and could enhance the degradation for electrocatalysis.

3.3.2. COD and TOC removal

Fig. 6 shows the result of the effect of potentials on color, COD and TOC removal ratios. For electrooxidative process, when the potential was lower than 600 mV, not only color removal ratio but also COD and TOC removal ratios were very low. It verified the inefficiency of electrosorption for the

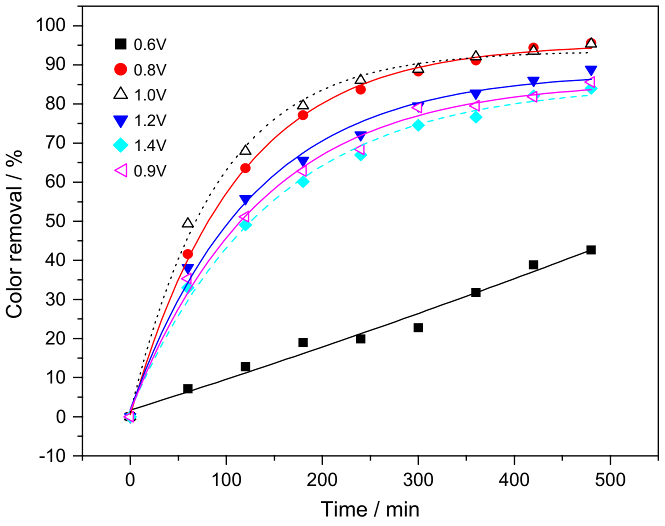


Fig. 5. Dependence of color removal on time for Amaranth solution on ACF with electrooxidation.

present system. When the potential was increased to 600 mV, though the color removal ratio increased dramatically, no remarkable increase in COD and TOC removal ratios occurred. When the potential was increased to 800 mV, the color removal ratio increased almost up to 95% and the COD and TOC removal ratios increased to 30% and 35%, respectively. The result implied that when the higher potential was given, the structure probably maybe destroyed, but not completely mineralized. Further increasing in potential after 800 mV, color removal ratio was almost constant, even decreased. COD and TOC removal ratios made little difference.

The results of UV-spectra of Amaranth at some positive potentials before and after treatment are shown in Fig. 7. As can be seen that at the potential of 800 mV, the UV-spectrum changed greatly and had a weak absorbance at 520 nm. It is confirmed that the cleavage of —N═N— was destroyed most completely at 800 mV. And the absorbances at 330 nm and 280 nm also decreased much.

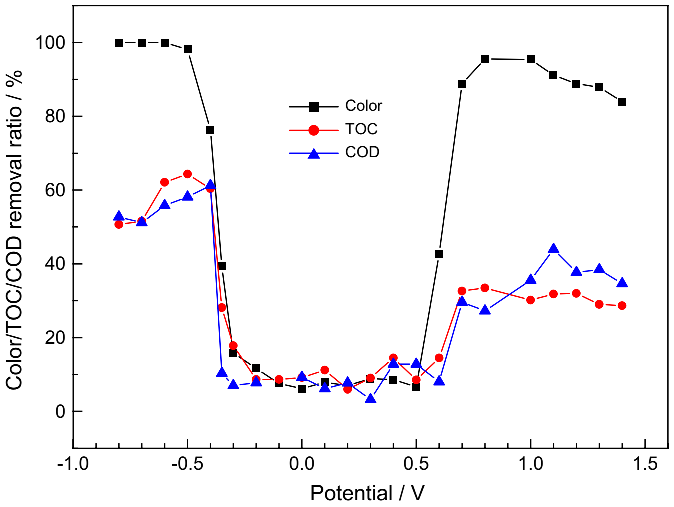


Fig. 6. The relationship between color, TOC, and COD removal ratios and potentials.

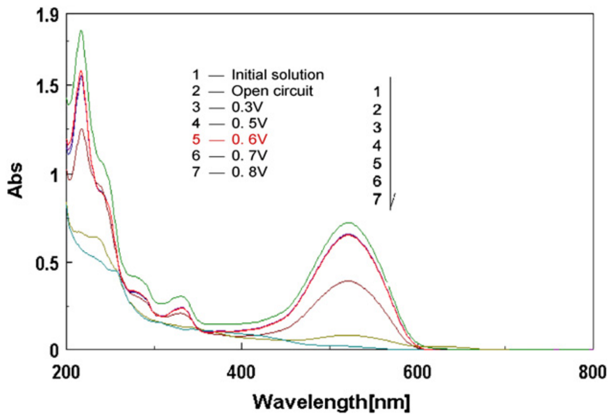


Fig. 7. UV–vis spectra of Amaranth solution at different positive potentials.

3.4. Process of electroreduction

3.4.1. Kinetics of the electrochemical process

The result of the color removal on time for electroreduction is shown in Fig. 8. It is clear that the higher the potential was in the range of −200 mV to −800 mV, the more the color removal was. At −200 mV, no obvious color removal was obtained. However, at −350 mV, the color removal ratio was almost 39.3% which was over 5 times of that under open circuit. Further by increasing the potential to −600 mV, the color of the dye water was completely removed at the end of the test. If the potential was up to −800 mV, the color removal ratio can be 100% at the 5th hour.

3.4.2. COD and TOC removal

For the process of electroreduction shown in Fig. 6, the color removal ratio was more than 75% at the potential of −400 mV and the COD and TOC removal ratios were both up to 60%. Further increase in the potential results in the color

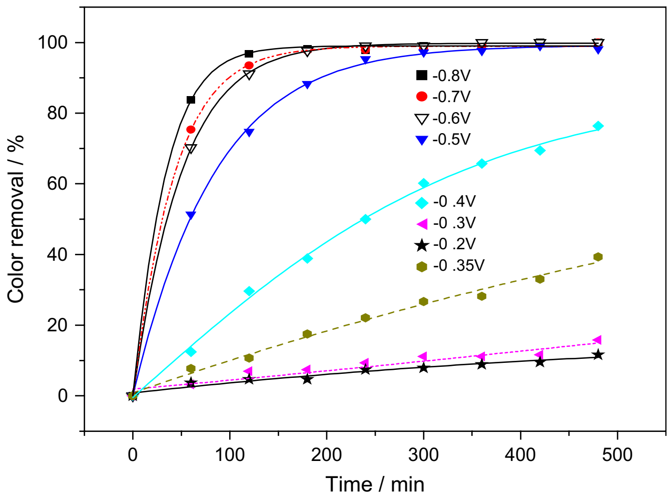


Fig. 8. Dependence of color removal on time for Amaranth solution on ACF with electroreduction.

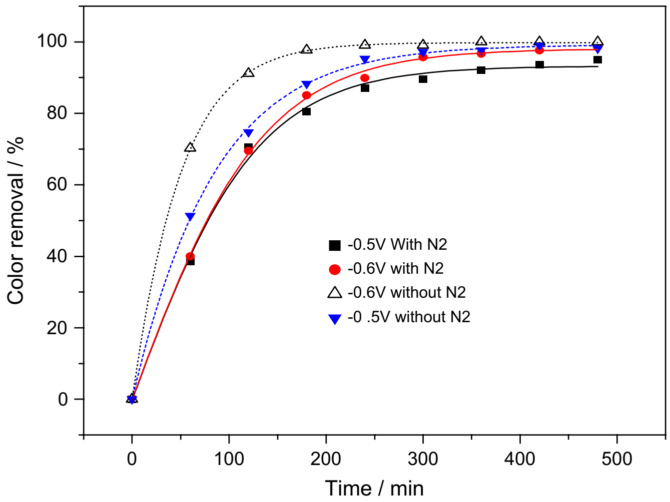


Fig. 9. Color removal of Amaranth at the potential of −0.5 V and −0.6 V on ACF by comparison of with and without N_2 during the electrochemical reduction.

removal ratio of 100%. For the COD and TOC removal ratios, they were almost constant at the value obtained at −400 mV.

Based on the average sense that the process of electroreduction cannot remove COD. However, the removals of COD were even much more than those in the process of electrooxidation. In order to make sure more how the electroreduction works, an airproof set-up was designed in order to avoid O_2 entering the set-up. The experiment of electroreduction of Amaranth aqueous solution on ACF was made with N_2 during the reaction. It is known that −0.5 V and −0.6 V are the preferred potential for H_2O_2 to be produced. So the two potentials were tested with N_2. In this condition, there is no O_2 exited and H_2O_2 could not be produced. The result of COD removal is shown in Fig. 9. From this figure we can see that the results for the COD removal with or without N_2 were almost the same. So the decolorization did not result from the H_2O_2 production on ACF. A conclusion could be made that the removals of color and COD resulted from the electroreduction of Amaranth and the absorption of the products on the ACF.

The results of UV-spectra of Amaranth at some negative potentials before and after treatment are shown in Fig. 10. At the potential of −800 mV, there is almost no absorbance in the visible range. Also, the absorbency values during UV region decreased sharply compared with the initial solution. It confirms that it is an efficient system to reduce the azo dye.

4. Conclusions

The potentiostatically electrochemical degradation with ACF as working electrode for azo dye Amaranth aqueous solution was examined with satisfactory results. The mechanism of the electrochemical processes was defined as adsorption, electrooxidation and electroreduction. The electrooxidation potential and electroreduction potential for the dye on ACF electrode were ascertained at 600 mV and −200 mV, respectively. The adsorption was insignificant for the removal of

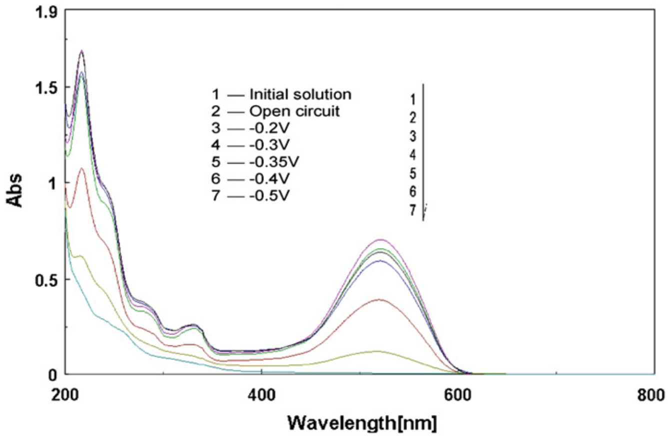


Fig. 10. UV–vis spectra of Amaranth solution at different negative potentials.

color, COD and TOC. The electrooxidation and electroreduction can both benefit the color, COD and TOC removal and electroreduction is better than electrooxidation. For the process of electrooxidation, the maximum color removal ratio obtained was about 95.6% with the COD and TOC removal ratios about 30%. For the process of electroreduction, the dye with the initial concentration of 80 mg L^{-1} was able to be completely removed after 5 h. At the same time, COD and TOC contents were both reduced by about 60%. The azo bond −N=N− was destroyed in the range of −800 mV to −200 mV and 600 mV–1400 mV.

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