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Electrochemical Advanced Oxidation of Acid Red Solution Using Carbon Felt or Glassy Carbon Cathode and Pt Anode

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ABSTRACT

In order to find an effective decolorization method for dye wastewaters, the present work aims at studying the treatment efficiency of an azo dye Acid Red 14 (AR14) by Electro-Fenton process using an undivided electrochemical cell containing different electrode materials. The optimal removal efficiency was obtained using carbon felt or glassy carbon (cathode) and platinum (anode) electrodes. The method is based on the reaction of electrochemically produced hydroxyl radicals leading to oxidative degradation of the AR14. To find the best conditions for treatment of AR14 dye, the effects of Fe^{2+} concentration, current density, the effect of pH initial, and the nature of support electrolyte were studied. The results showed 94 % removal efficiency in 30 minutes with 120 mA/cm^2 of electrolysis current, 0.2 mM of Fe^{2+}, and pH = 3. However, the decolorization efficiency measurements confirmed that the Electro-Fenton process with the platinum anode and the carbon felt cathode was more efficient.

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INTRODUCTION

The wastewater containing dyes is generally toxic, resistant to biodegradation, persistent in the environment, and challenging to treat using traditional oxidation processes. Advanced oxidation processes (AOPs) provide an effective means of rapidly treating compounds with efficient process control (Kayan et al., 2010).

The advanced oxidation processes (AOPs) based on the in situ production of a strong oxidant, mostly the hydroxyl radical (•OH), are considered promising efficient methods for azo dye removal from water (Wakrim et al., 2018). Chemical, photochemical, and electrochemical processes can all be used to generate hydroxyl radicals (Usman et al., 2018). The most popular AOP is the Fenton method, where a mixture of Fe^{2+} and H_2O_2 (Fenton's reagent) is used to degrade azo dye (Zhang et al., 2022). The electro-Fenton process, which is based on the continuous electro generation of H_2O_2 at a suitable cathode fed with O_2 or air, along with the addition of an iron catalyst to the treated solution to form the oxidant •OH as follows Fenton's

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reaction (Wang, 2008; Mohajeri et al., 2019; Sun et al., 2021; Wang et al., 2022).

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + HO^{\bullet} \quad k = 63\ mol^{-1}\ L\ s^{-1} \quad (1)$$

In contrast to the Fenton reaction, the Fe^{2+} and H_2O_2 are produced simultaneously by the reduction of the oxygen and ferric ions at the cathode via the following reactions (Bai et al., 2019; Divyapriya et al., 2020).

$$O_2 + 2H^+ + 2e^- \rightarrow H_2O_2 \quad E° = 0.69V/SHE \quad (2)$$

$$Fe^{3+} + e^- \rightarrow Fe^{2+} \quad E° = 0.77\ V/SHE \quad (3)$$

The cathode materials favoring electrogeneration of hydrogen peroxide H_2O_2 are gas diffusion electrodes (GDEs) (Panizza et al., 2009; Garcia-Segura et al., 2011; Yu et al., 2015; Bedolla-Guzman et al., 2016), graphite (Yuan et al., 2006; Nidheesh et al., 2014), and three-dimensional electrodes such as carbon-felt (Pimentel et al., 2008; Diagne et al., 2014; Olvera-Vargas et al., 2014), activated carbon fiber (ACF) (Lei et al., 2010; Wang et al., 2010), reticulated vitreous carbon (RVC) (Xie et al., 2006; Martínez et al., 2009), and carbon sponge (Özcan et al., 2008).

The main purpose of the present research is to study the ability of the electro-Fenton process to remove Acid Red 14 (AR14) dye from aqueous solutions using a two-electrode undivided cell configuration. Platinum (Pt) was used as the anode material for carbon felt and glassy carbon electrodes were compared as the cathode material. Some experimental parameters that affect the decolorization process such as the initial pH, Fe^{2+} concentration, electrolysis current, and the type of anion have been investigated to determine the optimum treatment conditions.

MATERIALS AND METHODS

Chemicals

The Acid Red (AR14) was the synthetic organic dye. Ferrous sulphate heptahydrate ($FeSO_4$, $7H_2O$), sodium sulfate (Na_2SO_4), and Sulphuric acid (H_2SO_4). All the chemicals used in the experiments were of laboratory reagent grade and used as received without further purification. All the solutions were prepared from distilled water and experiments were conducted at room temperature. The pH was measured using a HANNA HI8519N pH meter.

Electro-Fenton setup

Treatment of AR14 by the Electro-Fenton process was performed in an undivided electrochemical cell equipped with two electrodes (Figure 1). The working electrode was either a carbon felt piece (2*3*0.5 cm^3), a flexible material, resistant and easy to handle, or a glassy carbon 0.07 cm^2. The counter electrode was a platinum 14 cm^2. The applied current between these electrodes was available by a Potentiostat VoltaLab type PGZ 100. Prior to the electrolysis, compressed air was bubbled for 10 min through the cell to saturate the aqueous solution in oxygen. A concentration of sodium sulfate 1M (Na_2SO_4) was added to the solution as the supporting electrolyte. The iron sulfate ($FeSO_4$, $7H_2O$) catalyzing the Fenton reaction was added to the reaction medium before the beginning of the electrolysis. The pH of solutions was adjusted to 3 by sulphuric acid (10^{-3} M) to avoid the precipitation of ferric ions in the form of hydroxides. The initial concentration of AR14 dye used in this study was 1g/L. The samples were taken manually by a micropipette at definite time intervals and the spectroscopic experiments were carried out at λ = 515 nm using on Shimadzu dual-beam Spectrophotometer model UV-1800.

The removal efficiency was evaluated using relation (4)

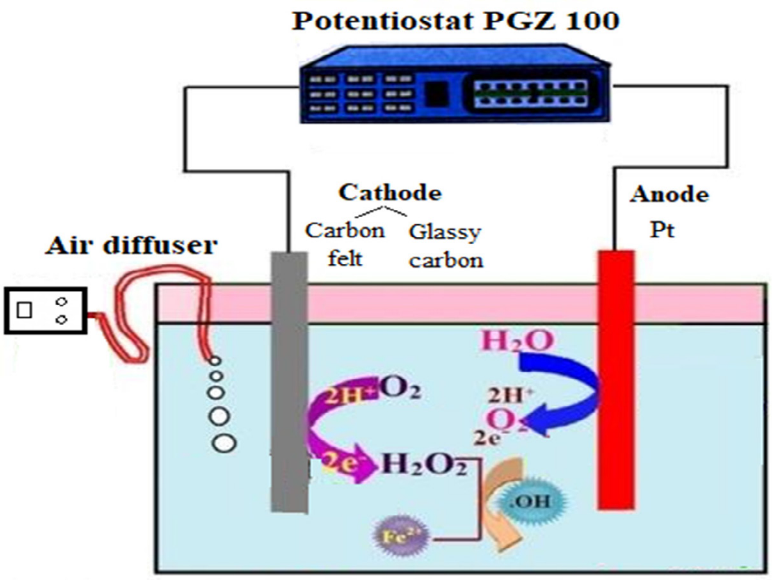

Fig.1. Experimental set-up for Electro-Fenton experiments

$$\text{Removal efficiency (\%)} = \left[\frac{Abs_0 - Abs_t}{Abs_0}\right] * 100 \quad (4)$$

Where Abs_0 and Abs_t are the initial absorbance and the absorbance at a time (t) of the treatment respectively.

RESULTS AND DISCUSSION

Decolorization of the AR14 solution using glassy carbon cathode

Effect of Fe^{2+} concentration

The effect of the Fe^{2+} concentration on the quality of the decolorization was studied at pH=3, with the electrolysis current of 200 mA/cm^2. The results are shown in Fig. 2.

The examination and analysis of Fig.2 illustrated that the decolorization efficiency of the AR14 solution was enhanced by increasing the concentration of Fe^{2+}. When the latter increased from 0.1 to 0.6 mM, the decolorization efficiency rose from 45% to 71% after 240 min of electrolysis time. A small amount of brown sludge, probably iron hydroxides, began to appear in the vicinity of the cathode when the Fe^{2+} concentration exceeded 0.4 mM. The presence of a layer of iron oxide on the cathode surface was observed probably because of the local alkalinization induced by H_2 evolution, as suggested by other authors (Petrucci et al., 2016). We subsequently fixed Fe^{2+} concentration at 0.2 mM to avoid the formation of the brown sludge.

Effect of electrolysis current

In the Electro-Fenton process, the applied current is an important parameter for operational cost and process efficiency (Hammami, 2008). To study the effect of applied current on the decolorization efficiency of AR14 solution, several experiments were carried out at different electrolysis currents in the presence of 0.2 mM of Fe^{2+}. The results are shown in Fig 3.

Analysis of Fig. 3 revealed that the decolorization efficiency increased with the electrolysis time and current density electrolysis. When the current density increased from 40 to 200 mA/cm^2, the decolorization efficiency of the AR14 solution increased from 43% to 68% at 240 minutes of

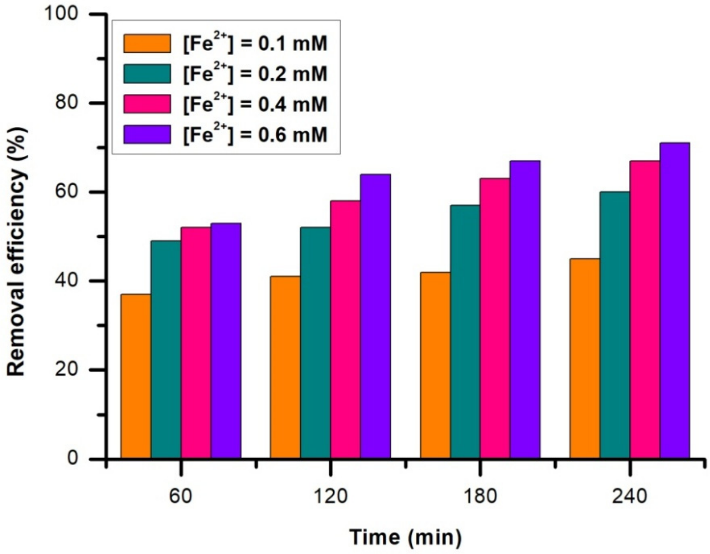

Fig. 2. Effect of Fe^{2+} concentration on the removal efficiency of AR14 solution, [Dye] = 1 g/L; i = 200 mA/cm^2; pH_i = 3; bubbled O_2

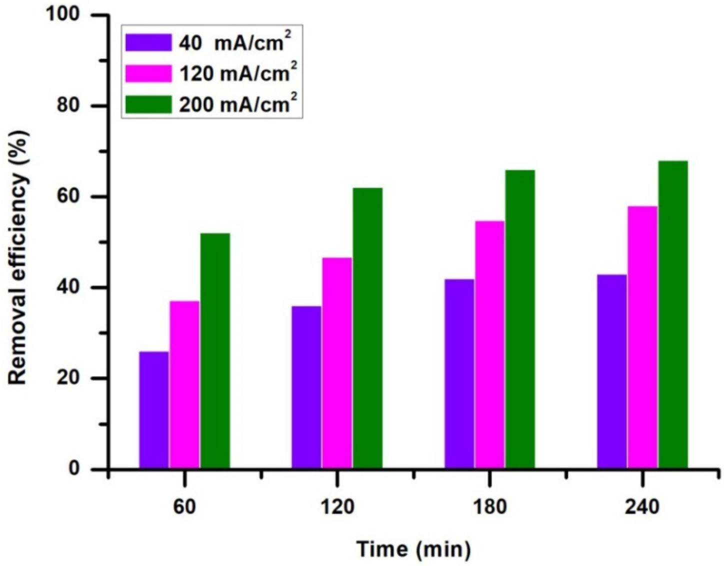

Fig.3. Effect of electrolysis current on the removal efficiency of AR14 solution. [Dye] = 1 g/L; [Fe^{2+}] = 0.2 mM; pH_i = 3; bubbled O_2

electrolysis time. We observed that the improvement in decolorization of the electrolysis current density was not significant at 200 mA/cm^2.

Decolorization of the AR14 solution using carbon felt cathode

The nature of the electrodes likely influenced decolorization efficiency. In this part of the work, tests were carried out using carbon felt cathode and platinum anode.

Effect of electrolysis current

Different solutions with the same AR14 concentrations (1g/L) were electrolyzed at different current densities ranging from 40 to 200 mA/cm^2 using carbon felt as a cathode. The results are presented in Figure 4. When the electrolysis current increased from 40 to 120 mA/cm^2, the removal efficiency of AR14 was improved. This was due to a higher production of the Fenton reagent (Fe^{2+}/H_2O_2), which leads to a higher production of hydroxyl radicals.

However, the continuous increase of applied current from 120 mA/cm^2 to 200 mA/cm^2 didn't result in the remarkable enhancement in AR14 removal rate correspondingly. The AR14 removal efficiency was slightly increased and reached 94% when the applied current was 200 mA. Consequently, 120 mA/cm^2 was considered the optimal applied current for AR14 degradation in this research. Current intensity could affect the generation efficiency of H_2O_2 at the surface of the cathode. It was reported that higher current intensity will lead to the formation of more H_2O_2 (Eq. (2)), subsequently, more $^\bullet OH$ will be generated through homogeneous EF reaction (Eq. (5)) and surface catalyzed process, and the increased current could also speed up the regeneration process of Fe^{2+} (Eq. (3)) (Du et al., 2020). The generated large amounts of $^\bullet OH$ could accelerate the degradation of AR14 in aqueous solution.

$$Fe^{2+} + H_2O_2 + H^+ \rightarrow Fe^{3+} + H_2O + {}^\bullet OH \quad (5)$$

Effect of the initial pH

The formation of the hydroxyl radical and the concentration of ferrous ions are both controlled by the pH of the solution. Hence, pH is an important parameter in the electro-Fenton process. The influence of initial pH (2, 3, 5, 7, 9) on AR14 removal efficiency was studied. Increasing the pH from 2 to 3 resulted in a slight increase in removal efficiency, as shown in Fig. 5. However, as the pH was increased from 3 to 9, the removal efficiency decreased from 96% to 59%. Therefore, pH=3 was determined as the optimum pH. This is because in acidic conditions and because of the simultaneous presence of hydrogen (H^+) and ferrous (Fe^{2+}) ions, the ideal conditions are provided for performing the Fenton process so that it is able to degrade hydrogen peroxide and produce the greatest concentration of hydroxyl radical. Similar results have already been reported (Yao et al., 2022). The diminished removal efficiency at a pH lower than 3 can be attributed to the fact that at a very acidic pH (lower than 3), hydrogen ions act as

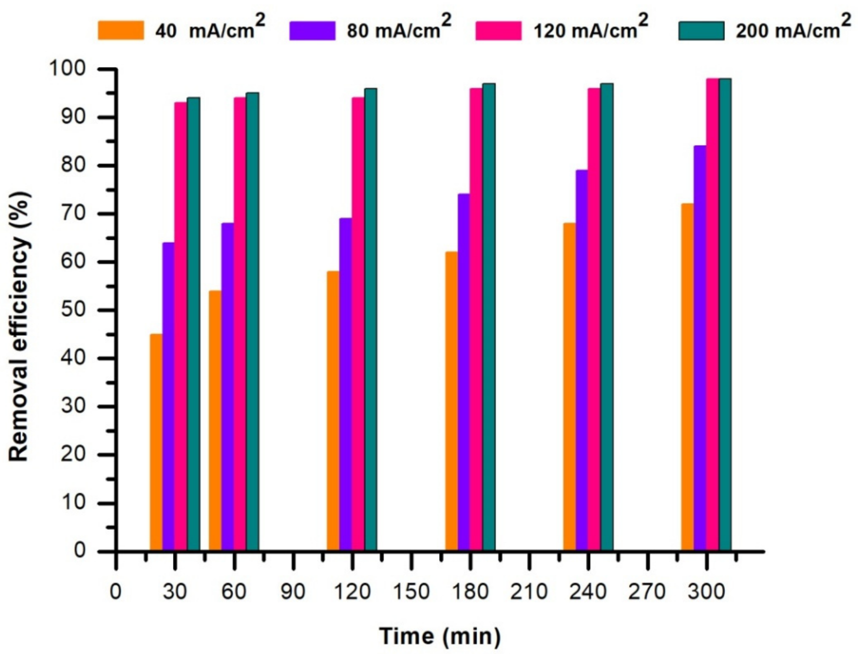

Fig.4. Effect of electrolysis current on the removal efficiency of AR14 solution. [Dye] = 1 g/L; [Fe^{2+}] = 0.2 mM; pH_i = 3; bubbled O_2

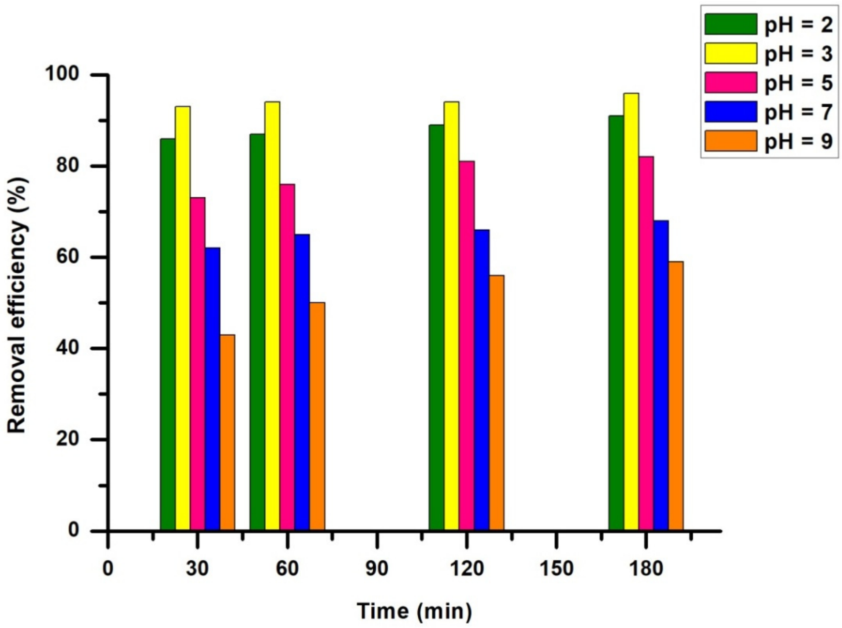

Fig.5. Effect of initial pH on the removal efficiency of AR14 solution. [Dye] = 1 g/L; $[Fe^{2+}]$ = 0.2 mM; i = 120 mA/ cm^2, bubbled O_2

Table 1. Effect of the initial pH in the electro-Fenton process in some studies

No	Pollutant	Electrodes	Optimum pH	Efficiency (%)	Time (min)	Ref
1	Ciprofloxacin	LDH/CF DSA	3	91.30	90	(Yao et al., 2021)
2	real dyeing wastewater.	activated carbon fibe/Pt	3	75.2	240	(Wang et al., 2010)
3	Alizarin red	gas- diffusion cathodes /Pt	3	93	240	(Panizza et al., 2009)
4	cationic red X-GRL	$ACF/RuO_2/Ti$	3	97	180	(Lei et al., 2010)
5	wastewater (ML-GFW)	iron electrode plates	3	71.9	180	(Wang et al., 2019)
6	Rhodamine B	Graphite	3	>90	180	(Nidheesh et al., 2014)

electron grabbers, causing the conversion of hydroxyl radicals into water Reaction (6) (F Javier Benitez, Francisco J Real, Juan L Acero, 2007).

$$OH^{\bullet} + H^{+} + e^{-} \rightarrow H_2O \quad (6)$$

At pH above 5, ferric ions precipitate as ferric hydroxide ($Fe(OH)_3$) and ferric oxyhydroxide ($FeOOH^{2+}$) and leave the Fenton reaction. The electro-Fenton process nearly stops when the pH rises to around 9 and is replaced by the coagulation process. As a result, efficiency decreases further (Nidheesh et al., 2012).

Table 1 depicts more information on the effect of the initial pH in the electro-Fenton process for the removal of persistent organic pollutants from water/wastewater.

Effect of the anion type

The effect of the anion nature (Cl^-, SO_4^{2-}, NO_3^- and $CH_3CO_2^-$), on the decolorization efficiency of AR14 dye, has been studied. The results are presented in Figure 6.

Examination of figure 6 indicates that using $NaNO_3$ or $NaCH_3CO_2$ as support electrolyte showed that the decolorization efficiency reached a maximum of 50% after 60 min of electrolysis time. But, using Na_2SO_4 and NaCl as support electrolyte-enhanced significantly the decolorization efficiency. It reached more than 95% during the same electrolysis time. This behavior can be explained by the competitive oxidation effect of $^{\bullet}OH$ radicals and chlorine-based oxidants such as Cl_2, HClO, and/or ClO^- electro generated at the anode as follows (Hassan et al., 2018):

$$Cl + H_2O + 2e^- \rightarrow HClO + H^+ + Cl^- \quad (7)$$

$$HClO \rightarrow ClO^- + H^+ \quad (8)$$

HClO is the predominant species formed at pH=3 (Malakootian et al., 2017).

On the other hand, between 120 and 180 min of treatment, a decrease in the decolorization rate was observed (85%). This may be due to the reaction between chloride ions and hydroxyl radicals $^{\bullet}OH$ to form the $Cl^{\bullet}$ radicals, which inhibited the reactivity of hydroxyl radicals and chloride ions according to the following reactions: (Nohara et al., 1997).

$$Cl^- + {}^{\bullet}OH \rightarrow ClOH^{\bullet -} \quad (9)$$

$$ClOH^{\bullet -} \leftrightarrow Cl^{\bullet} + OH^- \quad (10)$$

$$Cl^{\bullet} + Cl^- \rightarrow Cl_2^{\bullet -} \quad (11)$$

Therefore, based on these results and the literature studies, the sodium sulphate Na_2SO_4 can be used as the best support electrolyte (Brillas et al., 2003).

Comparison of the electrode nature on the removal efficiency of AR14 solution

Under the operating conditions used, i.e., dye concentration (1g/L), electrolysis current of

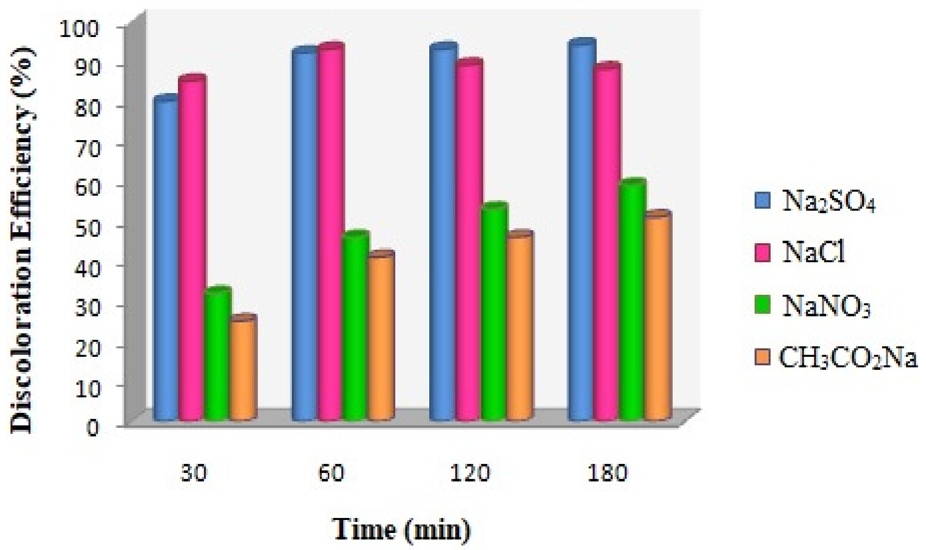

Fig. 6. Effect of the anion type on removal efficiency of AR14; [Dye] = 1 g/L, $[Fe^{2+}]$ = 0.2 mM, i =120 mA/cm^2, pH_i = 3

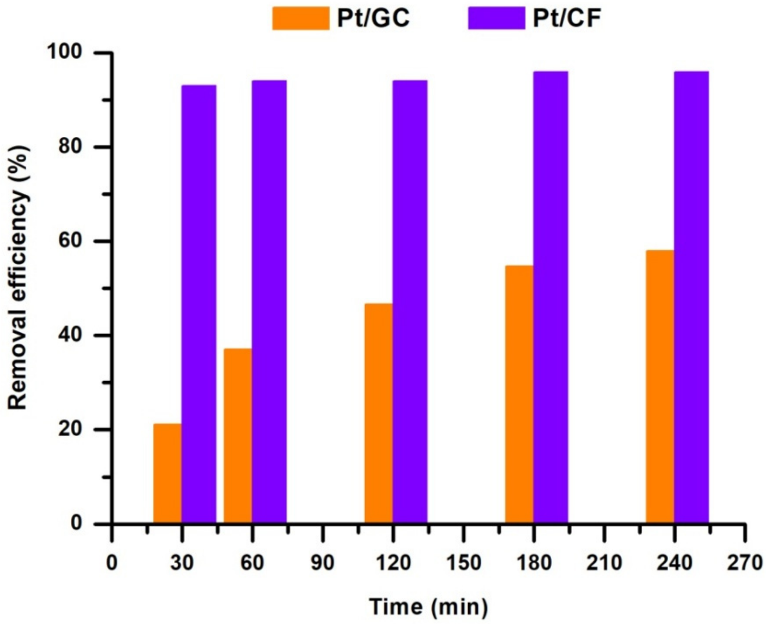

Fig. 7. Effect of the electrode nature on the removal efficiency of AR14 solution; [Dye] = 1 g/L, $[Fe^{2+}]$ = 0.2 mM, i = 120 mA/cm^2, pH_i = 3

120 mA/cm^2; and ferrous ion concentration 0.2 mM, the comparison of the removal efficiency of AR14 solution on glassy carbon (GC) and carbon felt (CF) cathode is presented in Figure 7.

The results of the decolorization efficiency measurements confirmed that the Electro-Fenton process with the platinum anode and the carbon felt cathode was more efficient. The decolorization efficiency of 94% of AR14 solution was achieved with the Electro-Fenton process after 30 min. On the other hand, with the glassy carbon cathode, the decolorization efficiency was less important.

According to the study carried out on the effect of the various operating parameters on the decolorization efficiency of AR14 solution by the Electro-Fenton process, we found that electrolysis current of 120 mA/cm^2 and initial concentration of ferrous ions 0.2 mM, proved to be the optimal operating conditions for decolorization of the AR14 solution.

Mechanism discussion

Based on studies, a possible catalytic degradation mechanism in the Electro-Fenton process with carbon felt cathode was proposed to comprehend the degradation process of AR14 during the reaction. As shown in Fig.8, H_2O_2 was generated through a two-step reaction at the carbon felt cathode. Firstly, O_2 was quickly transferred to the active site of the electrode through the porous structure, whereas O_2 rapidly produced $^{\cdot}O_2^-$ by capturing an electron (Eq. (12)). Next, $^{\cdot}O_2^-$ reacted with H^+ by trapping an electron and then electrochemical transformed into H_2O_2 (Eq. (13)). After that, H_2O_2 reacted with Fe^{2+} in the solution to produce •OH through Fenton reaction (Eq. (1)). Meanwhile, Fe^{2+} was regenerated through the electro reduction of Fe^{3+} (Eq. (3)). Anodic oxidation: AR14 adsorbed at anode surface could also be attacked by the electro-generated heterogeneous hydroxyl radicals (M(•OH)) (Eq. (14)) (Luo et al., 2020; Vasconcelos et al., 2022). •OH reacted with AR14 (Eq. (15)).

$$O_2 + e^- \rightarrow {}^{\cdot}O_2^- \quad (12)$$

$$^{\cdot}O_2^- + 2H^+ + e^- \rightarrow H_2O_2 \quad (13)$$

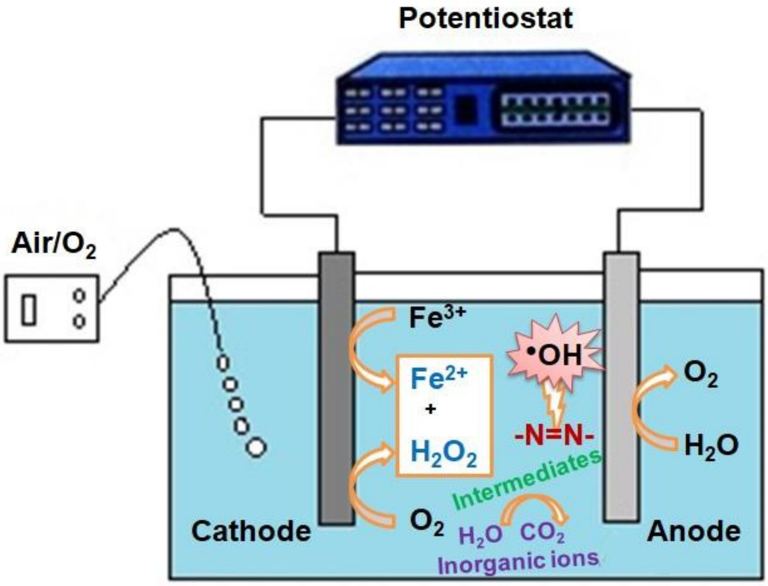

Fig. 8. Possible catalytic degradation mechanism for AR14 degradation in Electro-Fenton process

$$M + H_2O \rightarrow M(^{\bullet}OH) + H^+ + e^- \quad (14)$$

$$AR14 + {}^{\bullet}OH \rightarrow CO_2 + H_2O + \text{inorganic ions} \quad (15)$$

CONCLUSIONS

In this study, decolorization of Acid Red 14 (AR14) was investigated by electro-Fenton system using platinum as anode and glassy carbon or carbon felt as a cathode. The effects of parameters, namely Fe^{2+} concentration, electrolysis current, initial pH, and nature of support electrolyte were analyzed. The parameters determined to obtain an optimal 94 % removal efficiency of AR14 solution using carbon felt as cathode and Pt as anode were pH = 3, electrolysis current 120 mA/cm^2, the concentration of Fe^{2+} 0.2 mM, and 30 minutes of electrolysis time. It was concluded that decolorization of the azo dye AR14 solution was more efficient with carbon felt cathode compared to glassy carbon.

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The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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