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Fe (II) Immobilized on Activated Carbon Surface as a Catalyst for elimination of Alizarin Red S at the Presence of Hydrogen Peroxide

Hamzeh Saghafi

MSc of applied chemistry,
Department of chemistry, Semnan University, Semnan, Iran.

***Corresponding Author:**
h.saqafi@gmail.com

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ABSTRACT

In this study, removal of Alizarin red S(ARS) in aqueous solutions by oxidation process, using hydrogen peroxide (H_2O_2) and Fe(II) immobilized on activated carbon (AC) surface (Fe(II)/AC) as catalyst, was studied. In this case, the combination of catalyst and hydrogen peroxide significantly enhanced the removal process. The effects of pH, H_2O_2 and catalyst dosage, temperature and ARS concentration were also studied. The optimum conditions for ARS removal were obtained at pH=5, 2.5mL of 30% H_2O_2, 0.125gr of catalyst at 328K and 1×10^{-2}M of ARS concentration. In the optimum condition 99% of ARS was removed after 30min of reaction This work is considered as a suitable method for removal of high concentration of ARS in aqueous mediaOverall, the article highlights the potential of gold nanotechnology in developing effective and safe anti-aging strategies. Further research is warranted to explore the long-term safety and efficacy of nanoparticle-based formulations for skin rejuvenation and to optimize their delivery for enhanced therapeutic outcomes.

Keywords: Removal, ARS, Catalyst, H2O2, Hydroxyl Radical

Introduction

Dye Textile dyeing, one of the largest water consuming industries produces large volumes of wastewater in dyeing and finishing processes. Such effluents contain colored synthetic dyes and can pose a serious problem receiving water bodies. Moreover, dyes used in the textile industry may be toxic to aquatic organisms and can be resistant to natural biological degradation. Hence, the removal of color synthetic organic dyestuff from wastewater effluents becomes environmentally important [1]. There are more than 100,000 commercially available dyes with over 7×10^5 tons of dyestuff produced annually. Due to their chemical structure, dyes are resistant to fading on exposure to light, water, and many chemicals. Due to their complex structure and synthetic origin, many dyes are difficult to be decolorized and decomposed biologically. There are many structure varieties such as acidic, basic, disperse, azo, diazo, anthraquinone based, and metal complex dyes [2].

Alizarin Red S (1,2-dihydroxy-9,10-anthraquinonesulfonic acid sodium salt, ARS, Alias Mordant Red 3, C.I. no. 58005 is a water soluble, widely used anthraquinone dye. It is synthesized by sulfonation of alizarin which is a natural dye obtained from madder (Rubia tinctorum, L. Rubiaceae). In clinical practices, it is also used to stain synovial fluid to assess for basic calcium phosphate crystals. Anthraquinone dyes like ARS are durable pollutants, released especially by textile industries in the aquatic ecosystems. Removal of these from industrial wastewaters is a crucial process, from both economic and environmental points of view [3].

Several treatment processes are in practice for removal of dyes from wastewater processes include:

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adsorption on activated carbon [4,5], advance oxidation [6-12] membrane filtration [13,14], coagulation/flocculation [15,16], biological treatment [17,18] and etc.

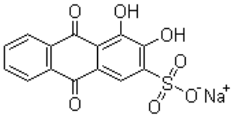
Advanced oxidation technologies (AOTs) are innovative methods for water treatment and are extremely useful in the case of substances resistant to conventional technologies. AOTs are oxidation processes which generate hydroxyl radicals (•OH) that are very effective in degrading organic pollutants because of their strong oxidant power ($•OH+H^+ +\bar{e} \rightarrow H_2O$; $E^0 = 2.80$ V) and non-selective species [19],[20]. O_3 /H_2O_2, O_3/UV, H_2O_2 /UV, TiO_2 /air/UV, Fe (II)/H_2O_2 (Fenton's reagent), Fe (III)/H_2O_2 (Fenton-like reaction), an oxidant (H_2O_2, O_3) and **Materials**
ultrasonic irradiation are the main types of AOTs that have been suggested in recent years [21-25]. Various combinations of them are employed for the complete mineralization of pollutants. Among these AOTs, Fenton's reagent is particularly attractive because of the low costs, the lack of toxicity of the reagents (i.e., Fe(II) and H_2O_2), the absence of mass transfer limitation due to its homogeneous catalytic nature and the simplicity of the technology [26].

In present work, the combination of Fe(II) and activated carbon as a catalyst on the removal of Alizarin S Red was studied. The results shown that the Fe(II)/AC was an influential catalyst for efficiency removal of ASR.

Experimental

In the present study, the anthraquinone dye, Alizarin Red S (ARS), was used for the removal process. Characteristics of ARS are present in table1. $FeSO_4$ was used as source of Fe(II), H_2O_2 solution 30%, H_2SO_4, NaOH, $Na_2S_2O_3$,ethanol and activated carbon were analytical grade and purchased from the Merck CO. Germany. The stock solutions were prepared by dissolving in distilled water.

Table 1. Chemical structure and characteristics of ARS

Molecular structure	Molecular formula	Molecular weight	C.I. number	synonyms	λ max
	$C_{14}H_7NaO_7S$	342.26	58005	9,10-Dihydro-3,4-dihydroxy-9,10-dioxo-2-anthracenesulfonic acid sodium salt	422 nm

Preparation of Fe(II)/AC catalyst

For preparation of catalyst, 5gr of activated carbon was stirred with phosphoric-acid (1M) at ambient temperature for activation of activated carbon surface for 6 hours [27]. Then the activated carbon washed with doubly distillated water until the acid wholly removed. After the activation of activated carbon surface, 5g of $FeSO_4.7H_2O$ was dissolved in minimum of doubly distillated water and was added to activated carbon. The mixture was dispersed in ethanol and the dispersion was heated on the heater for 12 hours on a sealed system. After the mention time was finished the catalyst washed with doubly distillated water and the excess of Fe(II) was removed. After each wash, the concentration of excess Fe(II) in filtrate was determined by atomic absorption spectrometry(model shimadzu 608). The wash process was repeated in many times until that no more Fe(II) was detected in filtrate. Finally the amount of the Fe(II) immobilized on activated carbon surface was found 0.102gr by atomic absorption spectrometry. The Fe(II)/AC was dried at 328K for 6 horses. The SEM image of prepared catalyst is shown in Fig.1.

Experimental method

All of experiments were carried out in a batch system reactor, equipped by thermometer and mixer. To start of each test, 50 mL of dye solution was injected into reactor, then for the optimization of experimental conditions, pH of dye solutions was adjusted by 0.1M H_2SO_4 and 0.1M NaOH. The pH of dye solution was measured by pH meter model pH 462-TS technology. Amounts of 0.33-1.5gr of catalyst and H_2O_2 in volumes of 1.5-3mL were poured into dye solutions by mixing, then the reactor was sealed.
Temperature was controlled at range of 298–328K and measured by thermometer. Before sampling, the process of removal was blocked by adding of $Na_2S_2O_3$ and suspensive samples were centrifuged.

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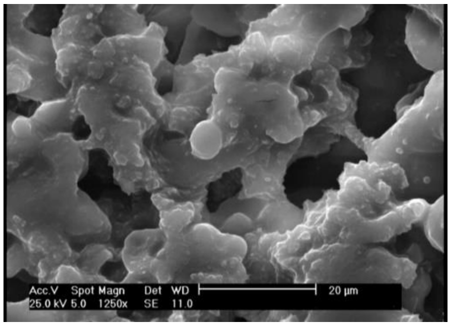

Fig.1. SEM image of Fe(II) immobilized on activated carbon surface

Furthermore, the samples were determined by UV-spectrophotometer model Shimadzu at 422nm in quartz cells. For comparative purposes, experiment was carried out separately, by AC, H_2O_2, AC/ H_2O_2, Fe(II)/ H_2O_2 under the optimum condition. The removal efficiency (%) of ARS is defined as follows:

$$\text{removal efficiency } (\%) = \left[1 - \left(\frac{C_t}{C_0}\right)\right] \times 100 \quad (1)$$

Where C_0 (mg L^{-1}) is the initial concentration of ARS and C_t (mg L^{-1}) is the concentration of ARS at reaction time, t (min).

Reaction mechanism description of catalyst at presence of H_2O_2

The AC/Fe(II) as a catalyst is included 2 main factor to enhance the removal process: activated carbon and Fe(II) for generation of •OH at presence of H_2O_2. The mechanism is based on the generation of •OH radicals by the catalytic decomposition of H_2O_2 in acidic media. In presence of Fe^{2+} and AC, the peroxide breaks down to •OH and OH^-, as the equations 2 and 3.

$$\mathrm{Fe(II)} + H_2O_2 \rightarrow \mathrm{Fe(III)} + \bullet OH + OH^- \quad (2)$$

$$\mathrm{AC} + H_2O_2 \rightarrow \mathrm{AC}^+ + \bullet OH + OH^- \quad (3)$$

Then Fe(III) and AC^+ can convert into Fe(II) and AC according to eqs.4-6.

$$\mathrm{Fe(III)} + H_2O_2 \rightarrow \mathrm{Fe(II)} + \bullet OOH + H^+ \quad (4)$$

$$\mathrm{Fe(III)} + \bullet OOH \rightarrow \mathrm{Fe(II)} + H^+ + O_2 \quad (5)$$

$$\mathrm{AC}^+ + H_2O_2 \rightarrow \mathrm{AC} + \bullet OOH + H^+ \quad (6)$$

Further increase in H_2O_2 dosage has negative influence due to the recombination of •OH radical and H_2O_2, contributing to the •OH scavenging capacity.

$$\bullet OH + H_2O_2 \rightarrow \bullet OOH + H_2O \quad (7)$$

$$\bullet OH + \mathrm{Fe(II)} \rightarrow \mathrm{Fe(III)} + OH^- \quad (8)$$

$$\bullet OH + \bullet OH \rightarrow H_2O_2 \quad (9)$$

$$\bullet OH + \bullet OOH \rightarrow O_2 + H_2O \quad (10)$$

The hydroxyl radical attacks on dye molecule and abstracts a hydrogen atom or adds itself to double bonds. In fact, Hydroxyl radical can rapidly and non-selectively react with a wide range of organic pollutants through hydrogen abstraction, electrophilic addition, and/ or electron transfer to mineralize them to CO_2, H_2O and inorganic ions [28].

Results

Effect of initial pH on the removal of ARS

pH value plays a significant role in the procedure of ARS elimination. The effect of pH for the removal of ARS was studied in the range of 2-6. The pH of ARS solutions was adjusted at the required value by adding 0.1M of H_2SO_4 or 0.1M of NaOH. The results were shown in Fig. 1. It could be seen in Fig. 1 at initial pH 2,3 the removal efficiency of ARS was not noticeable, because, H_2O_2 can stay stable probably by solvating proton to form an oxonium ion(i.e. $H_3O_2^+$), so the generation of •OH is restricted.

$$H_2O_2 + H^+ \rightarrow H_3O_2^+ \quad (11)$$

$$\bullet OH + H^+ \rightarrow H_2O \quad (12)$$

At the same time, the formed complex species $[\mathrm{Fe}\ (H2O)_6]^{2+}$ and $[\mathrm{Fe}\ (H2O)_6]^{3+}$ also reacted more slowly with H_2O_2. In addition, the scavenging of •OH by the excessive of H^+ was also another reason for the lower removal efficiency of ARS at pH 2,3 [29-31]. At pH 6 the removal of ARS was decreased too. It is principally due to the formation of ferrous/ferric hydroxide complexes lead to the deactivation of Fe(II)/AC catalyst, which results in the low generation of •OH. At pH 4 and 5 the removal process of ARS were increased. At pH 4 the removal efficiency of ARS after 75 min was achieved 90%, and 92% at pH 5.

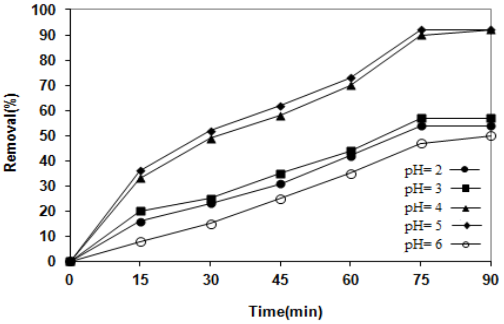

Fig.2. The effect of pH on the removal of ARS (0.33×10^{-3} M) in presence of presence of 1.5mL 30% H_2O_2 and 0.033gr of catalyst at 298 K.

Effect of H_2O_2 dosage on the removal of ARS

H_2O_2 is the main source for generation of •OH on the removal of ARS by catalyst process. The effects of 30% H_2O_2 dosages from 1.5mL to 3mL were studied

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and the results were shown in Fig. 2. From on the Fig. 2. It is shown that, the increasement of H_2O_2 from 1.5mL to 2.5mL can enhance the removal of ARS from 92% to 96% within 75 min of reaction. Obviously, by increasing of H_2O_2 dosage in the process, the generation of •OH was enhanced, so the ability of •OH on the removal process was improved. Furthermore, the removal of ARS was decreased from 96% to 80% by increasing of H_2O_2 from 2.5mL to 3mL, because at higher H_2O_2 dosage, the scavenging and recombination of •OH radicals will occur, so more radicals will be formed which can be expressed by the eq. 7, 9, 10.

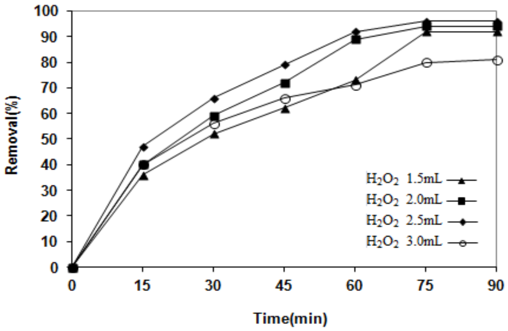

Fig.3. The effect of H_2O_2 dosage on the removal of ARS (0.33×10^{-3} M) at pH 5 in presence of 0.033gr catalyst at 298 K.

Effect of catalyst on the removal of ARS

Fe(II) and AC are two main agent for suitable removal of dyes at percent of H_2O_2 for generation of •OH(eq. 1,2). The effect of Fe(II) /AC as a catalyst on the removal of ARS with different amount from 0.033 to 0.125gr was studied and the results were shown in Fig. 3. The results indicated that the removal of ARS was significantly influenced by the amount of catalyst and the optimum amount was observed at 0.125gr of catalyst by the removal efficiency of 97%. the results for removal of ARS at 0.033gr was obtained 92% after 60 min of reaction time. The removal efficiencies of ARS were improved in the presence of 0.066, 0.1 and 0.125gr of catalyst after 60 min by 93%, 95% and 97% respectively. In addition, at low amount of catalyst the removal efficiency of ARS was limited because the catalyst couldn't able to generated of •OH, but higher removal of ARS efficiency achieved at high amounts of catalyst was mainly attribute to the higher production of •OH with more catalyst in the removal process at low time so the concentration of •OH in the reaction media for suitable removal of ARS was increased.

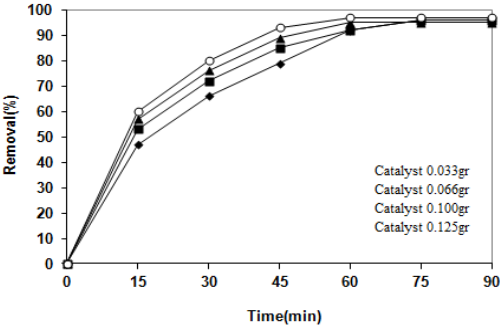

Fig .4. The effect of catalyst amount on the removal of ARS (0.33×10^{-3} M) at pH 5 in presence of 2.5mL 30% H_2O_2 at 298 K.

Effect of temperature on the removal of ARS

The temperature of reaction is another important parameter on removal of ARS process. The removal of ARS at different temperature 298 to 328K was studied and the results were shown in Fig.4. As can be seen from the Fig. 4, High removal of ARS was obtained at high temperature. In fact, the temperature exerted a strong effect on the removal efficiency of ARS and the removal was accelerated by a raise in temperature (298, 308, 318 and 328 K). This is because higher temperature increased the reaction rate between H_2O_2 and the catalyst, thus increasing the rate of generation of •OH. The removal efficiency after 45 min of reaction increased from 93%, 95%, 97% to 99% by increasing the temperature from 298, 308, 318 to 328K. Additionally, the period of required time for the removal of ARS was also much shorter at high temperature so, increasing the temperature has a positive impact on the removal of ARS. The optimum condition was selected at 318K for removal of ARS.

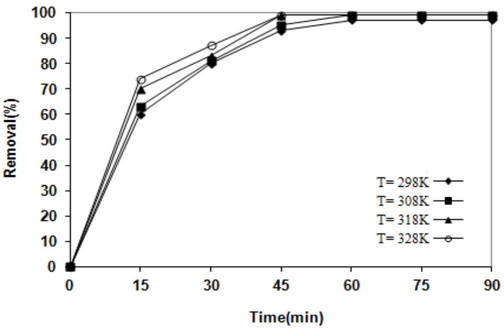

Fig. 5. The effect of temperature on the removal of ARS (0.33×10^{-3} M) at pH 5, in presence of 2.5mL H_2O_2 and 0.125gr of catalyst.

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Effect of concentration of ARS on the removal of ARS

The removal of ARS in different concentrations were studied, and the results were shown in Fig. 5. The results showed that the removal of ARS was decreased from 99% to 64% by increasing concentration of ARS from 0.33×10^{-3} to 1.5×10^{-2}M after 45 min. It was cleared that, at the presence of constant amount of catalyst and H_2O_2 and constant generation of •OH, the removal efficiency at high concentration of ARS showed a decrease rate on removal efficiency.

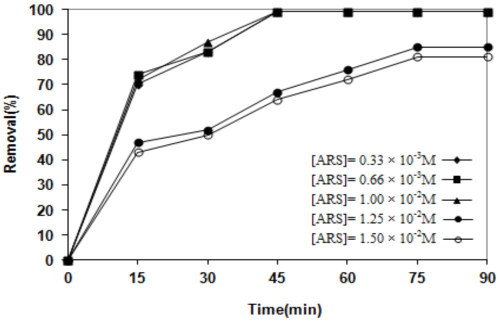

Fig. 6. The effect of dye concentration on the removal of ARS at pH 5, in presence of 2.5mL H_2O_2 and 0.125gr catalyst at 318 K.

The compare of removal process by Fe(II)/AC and other possible conditions

The compare of present work and other possible conditions for removal of ARS such as using AC and H_2O_2, Fe(II) and H_2O_2, H_2O_2, AC as catalysts and reagents under the optimum experimental results, were studied. The results were shown in Fig. 6. As can be seen from the Fig. 6, the removal efficiency at presence of H_2O_2 was very low. Because the H_2O_2 without catalyst couldn't able to generate •OH. The removal of ARS by AC was also limited, because the mechanism of ARS removal in this section was according to adsorption on the AC surface. When, Fe/H_2O_2 or AC/H_2O_2 was used as a catalyst for the removal of ARS, the removal reaction was lower than of using the studied catalyst and the results for removal efficiency were 92% and 94% after 75 min for Fe/H_2O_2 or AC/H_2O_2. In fact, the rate of generation of •OH in presence of H_2O_2 and Fe(II) or AC, was limited in comparison with Fe(II)/AC as catalyst.

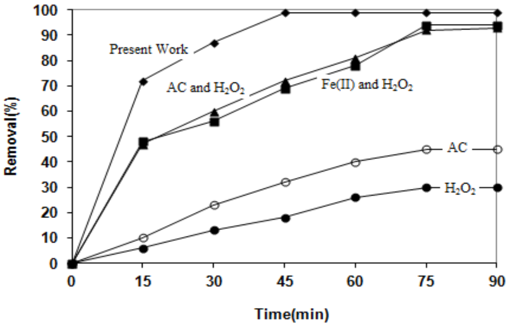

Fig. 7. The compare of removal process by the Fe(II)/AC as a catalyst and other conditions under the optimum experimental results at pH 5, in presence of 2.5mL H_2O_2 and 0.1×10^{-2}M of ARS, 0.125gr catalyst at 318 K .

conclusion

In this work, the removal of ARS in aqueous solution by Fe(II)/AC at presence of H_2O_2 has been studied and the present investigation draws the following conclusion:

(i) Removal of ARS by mixture of hydrogen peroxide and Fe(II)/AC, is capable of releasing hydroxyl radicals which may take part in oxidation of dissolved ARS in wastewater.

(ii) The optimal operation parameters for the removal of ARS were 0.1gr of catalyst, and 2.5mL of 30% H_2O_2 for 50mL, 1×10^{-2}M of dye concentration at pH=5 with 318K temperature.

(iii) Under the optimal conditions, 99% removal efficiency of ARS in aqueous solution was achieved within 45 min.

Another positive point is, AC is a stabilizer phase for Fe(II) and after oxidation with H_2O_2, the Fe(III) can't move into solutions media and AC is a suitable agent for the prevention of pollution with Fe(III) in this method.

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