ДЕСТРУКЦИЯ КРАСИТЕЛЯ КИСЛОТНОГО ОРАНЖЕВОГО-7 В ВОДНЫХ РАСТВОРАХ В ПРИСУТСТВИИ ИОНОВ ЖЕЛЕЗА (III), ПЕРСУЛЬФАТА ПРИ ВОЗДЕЙСТВИИ ВИДИМОГО ИЗЛУЧЕНИЯ

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В работе изучена деструкция красителя кислотного оранжевого-7 (АО7) в водных растворах при воздействии иона железа (III), активации процесса окисления ионом персульфата (PS, $S_2O_8^{2-}$) и воздействии видимого излучения (Vis) (≥420 нм). Проведена оценка эффективности различных систем, включая Fe^{3+}/Vis, Fe^{3+}/PS, PS/Vis и Fe^{3+}/PS/Vis. Установлено, что эффективность системы Fe^{3+}/PS и PS/Vis оказалась невысокой, при этом окислению подвергалось лишь порядка 48% АО7 в течение 20 мин под воздействием системы Fe^{3+}/Vis с последующим ростом эффекта до почти 95% в течение 20 мин под воздействием системы Fe^{3+}/PS/Vis. С целью оптимизации процесса изучено влияние на него таких технологических параметров, как концентрация PS, Fe^{3+}, красителя, а также значения начальной величины pH. В результате показано, что эффективность деструкции красителя увеличивается с ростом концентрации PS и Fe^{3+} в исследуемом диапазоне времени воздействия. Значение pH не оказывает существенного влияния на деструкцию АО7. Изучено также влияние присутствия в воде неорганических анионов, при этом показано, что их эффект ингибирования процесса может быть ранжирован в порядке возрастания: $Cl^-≈NO_3^- <SO_4^{2-} <HCO_3^-≈HPO_4^{2-}$.

Ключевые слова: разложение красителя, кислотный оранжевый 7, персульфат, фотокаталитическое окисление, видимый свет, неорганические отрицательные ионы

DEGRADATION OF ACID ORANGE 7 IN AQUEOUS SOLUTION UNDER PRESENCE OF IRON(III), PERSULPHATE AND VISIBLE LIGHT IRRADIATION

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In this study, the degradation of Acid Orange 7 (AO7) in aqueous solutions by iron(III) under activation by persulfate (PS, $S_2O_8^{2-}$) oxidation and Visible (Vis) light (≥420 nm) exposure was performed. The efficiency of different systems including Fe^{3+}/Vis, Fe^{3+}/PS, PS/Vis and Fe^{3+}/PS/Vis has been evaluated. It was found that the efficiency of Fe^{3+}/PS and PS/Vis system was low, and only about 48% of AO7 was oxidized within 20 min of experiment time under Fe^{3+}/Vis system action, and it was further improved to almost 95% within 20 min by Fe^{3+}/PS/Vis system. Various process-dependent parameters, such as PS, Fe^{3+}, dye concentration as well as initial pH were investigated to optimize the process. As results indicated, the dye's degradation efficiency increased with the increase of PS and Fe^{3+} concentration over the tested range. The pH had an insignificant effect on the degradation of AO7. The effect of inorganic anions was also investigated and the suppressive effects of inorganic anions can be ranked in an ascending order of $Cl^-≈NO_3^-<SO_4^{2-}<HCO_3^-≈HPO_4^{2-}$.

Key words: dye degradation, Acid Orange 7, ferric iron, persulfate, photocatalyst oxidation, visible light, inorganic negtive ions

INTRODUCTION

Advanced oxidation processes (AOPs) are considered as one of the most effective methods to degrade organic toxic contaminants. Recently, persulfate (PS, $S_2O_8^{2-}$) with a redox potential equal to 2.01 V [1], attracts high attention due to its easy storage and transport, high stability, high aqueous solubility, and comparatively low cost [2-3]. Extensive research works have been held on the activation of PS under: interaction of heat [4-5], base activation [6], ultraviolet (UV) light (sensitive wavelength 220-310 nm) [7-10] activation, ultrasound activation [11], and transition metals activation [12-17].

Although heat and UV are effective methods to activate PS, the intrinsic disadvantage of high cost and complexity limit their widespread practical application. Among the activators, Fe^{2+} has been commonly selected due to its great practical application's advantages, such as high efficiency, large abundance, low cost and environmental friendly nature [18, 19].

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However, the usage of PS/Fe^{2+} system is limited because of Fe^{2+} unstability in air and slow conversion from Fe^{3+} to Fe^{2+} [20-21]. Recently, to improve the conversion efficiency, Zou et al. discussed the method to degrade benzoic acid by adding hydroxylamine (HA) into Fe^{3+}/PMS process, during which the redox cycle of Fe^{3+}/Fe^{2+} was accelerated [22]. Heng Lin et al. reported that the clofibric acid degradated by an EC/Fe^{3+}/PMS process, and Fe^{2+} can be regenerated through cathodic reduction reaction [23]. Mavronikola C et al. investigated the decolorization of AO7 in the presence of iron(II) sulfite complex and oxygen under UV-vis irradiation, and found that ferrous ions could be regenerated under UV-visible irradiation [24] (Eqs. (1)).

$$Fe^{3+} + H_2O + hv\ (\lambda<450\ nm)\rightarrow Fe^{2+} + \cdot OH + H^+ \quad (1)$$
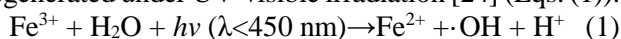
Compared with UV light, the Vis light, covering about 45% of the sunlight, as a cost-effective and environmentally friendly energy, has been used [25]. Zhao et al. investigated the application of Vis/Fe^{3+}/H_2O_2 treatment process to the aqueous degradation of cationic acridine orange monohydrochloride and anionic alizarin violet 3B, and confirmed that Fe^{2+} can be regenerated in the visible part of the spectrum by enhancing the photo reduction of Fe^{3+} to Fe^{2+} (Eqs. (2) and (3)) [26, 27].

$$Dye+hv\ (\lambda>470\ nm)\rightarrow Dye^* \quad (2)$$
$$Dye^*+Fe^{3+}\rightarrow Dye^+ + Fe^{2+}, \quad (3)$$
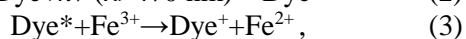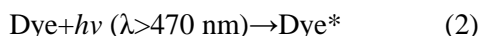
the sign "*" means here that dye is excited under irradiation of visible light and so it's at the high-energy state.

For further efficiency enhancement of the system Vis/PS/Fe^{3+}, a valuable approach, the matter of which is the combination of PS with Fe^{3+} under the irradiation of Vis light to degrade organic pollutants is provided.

As a representative compound of azo dyes, AO7 was selected for this study. The objective was to investigate the enhancement of the degradation efficiency of AO7 in Fe^{3+}/PS process under visible($\geq$420 nm) irradiation and specifically focused at: the activity for the system Fe^{3+}/PS/Vis, the effect of Fe^{3+}/PS/AO7 concentration, the effect of initial pH and the effect of inorganic negtive ions.

EXPERIMENTAL

1. Materials

Acid Orange 7 (AO7, $C_{16}H_{11}N_2NaO_4S$) was purchased from Aladdin Industrial Corporation. Iron (3+) nitrate nonahydrate ($Fe(NO_3)_3 \cdot 9H_2O$), sodium persulfate ($Na_2S_2O_8$) and other chemicals were of analytical grade if not noticed otherwise and were obtained from Sinopharm Chemical Reagent Co., Ltd. All chemicals were used as received without further purifica-

tion. The sample solutions were prepared using deionized water (Aquapro, USA) throughout the experiments.

2. Degradation procedures

All batch experiments were conducted in a 100 mL undivided glass beaker containing 50 mL solution at about 25ºC without irradiation or under irradiation by a direct exposure Vis light power supply (CHF-XM-500W) from Beijing Trusttech Co. (China). After the desired amounts of AO7 and PS in 50 mL of the aqueous solution were added into the reactor, the reaction was initialized by adding Fe^{3+}. Each reaction solution was constantly agitated by a magnetic stirrer (model 78-1, Hangzhou Instrument Motors Factory, China).

A stock solution of AO7 with the initial concentration (C_0) 20 mg/L was prepared freshly with deionized water before each run. Since PS is an acidic oxidant, the addition of PS led to a significant decrease of pH, and the experiment was conducted at acidic medium (pH 3.46, no adjustment). For studying the effect of solution pH on the rate of AO7 degradation, 0.1 M sulfuric acid (Pronolab, 98%, 1.84 g/cm^3) and 0.1 M sodium hydroxide (Quimitécnica, 30% w/v, 1.33 g/cm^3) were used to adjust the solution pH after PS was added into the solution. Before the beginning of reaction, PS solution and Fe^{3+} solution were added to the reactor.

3. Analysis

To monitor the degradation process of AO7, solution samples were taken out at predetermined time intervals and measured immediately on a METASH UV-5500PC spectrophotometer under the maximum absorption wavelength (484 nm) to record the temporal UV–vis spectral variations of the dye. The related reaction progress was monitored using the UV-vis absorption spectra of AO7 in terms of the percentage of photodegraded azo-dye, which was calculated from the following equation 4 (percentage of decolorization):

$$AO7 = (C_0 - C_t)/C_0 \cdot 100\%, \quad (4)$$

where C_t is the absorbance at 484 nm for AO7 at reaction time t and C_0 is the related initial absorbance.

RESULTS AND DISCUSSION

1. Degradation efficiency of AO7 under different systems

To evaluate the activities of different systems, 20 mg/L AO7 was treated for 20 min by PS alone, Vis alone, Fe^{3+}/PS process, Vis/PS process, Vis/Fe^{3+}/PS process and Vis/Fe^{3+}/PS process. As shown in Fig. 1, the efficiency of Fe^{3+}/PS and PS/Vis system was low, and the efficiency of Fe^{3+}/Vis system was much higher, which could be further enhanced by adding PS.
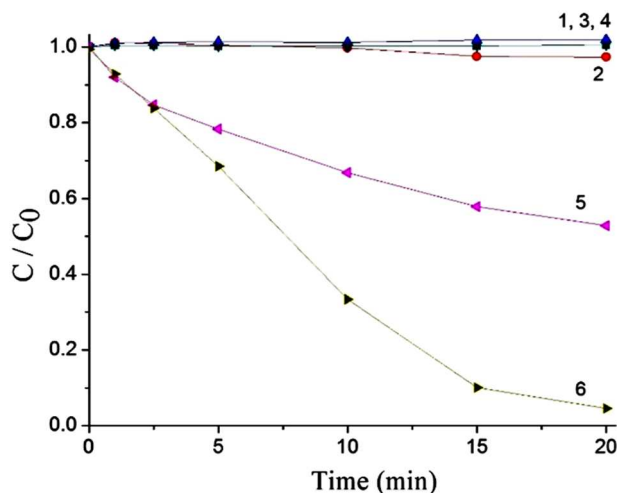
Fig. 1. Degradation of AO7 in different systems: (1) PS, (2) Fe^{3+}/PS, (3) Vis, (4) Vis/PS, (5) Vis/Fe^{3+}, (6) Vis/Fe^{3+}/PS
Рис. 1. Разрушение AO7 в различных системах. (1) PS, (2) Fe^{3+}/PS, (3) Vis, (4) Vis/PS, (5) Vis/Fe^{3+}, (6) Vis/Fe^{3+}/PS

Fig. 2. UV/Vis light absorption spectrum for AO7 by Vis/Fe^{3+}/PS process at different time (min): (1) 0, (2) 2.5, (3) 5, (4) 10, (5) 15, (6) 20
Рис.2. Спектр поглощения в УФ/видимой области кислотного оранжевого 7 при совместном действии света, Fe^{3+} и персульфата при различных временах (мин): (1) 0, (2) 2,5, (3) 5, (4) 10, (5) 15, (6) 20

About 48% of AO7 was oxidized under the system Fe^{3+}/Vis within 20 min, while the oxidation efficiency was significantly improved by Fe^{3+}/PS/Vis system: □95% of AO7 was oxidized in 20 min. Blank experiments (Fig. 1) showed almost no AO7 oxidation in the absence of Fe^{3+} or Vis. This phenomenon could be interpreted with the relative stability of PS at ambient temperature and its oxidation potential was very limited (E^0 = 2.01 V vs. NHE), which could hardly degrade AO7 via direct photolysis.

2. Spectral change of AO7

AO7 degradation was monitored in the experimental runs by means of UV/Vis scanning of the whole range of wavelengths available for a diode array and UV/Vis spectra were recorded at different reaction time in Fig. 2. As can be seen, the representative radiation absorption spectrum of AO7 in water is characterized by the band in the Vis region, with the maxima located at 484 nm, corresponding to the n-π* transition of the azo form (the sign "*" means here the antibinding orbitals), and by two bands in the ultraviolet region located at 262 nm and 308 nm, attributing to benzene-like structures in the molecule, and also a band at 214 nm, considered to be associated with Fe(III). It is observed that the intensity of the signal at 484 nm becomes smaller as the irradiation time increases until no signal is detected in the analyzed wavelength range, indicating the destruction of azo chromophore. At the meantime, the bands at 262 nm and 308 nm are disappeared due to the fragmentation of "benzene-like" structures. No additional UV/Vis signals were detected in the sample analysis, meaning that no radiation absorbing intermediates at this wavelength were generated.
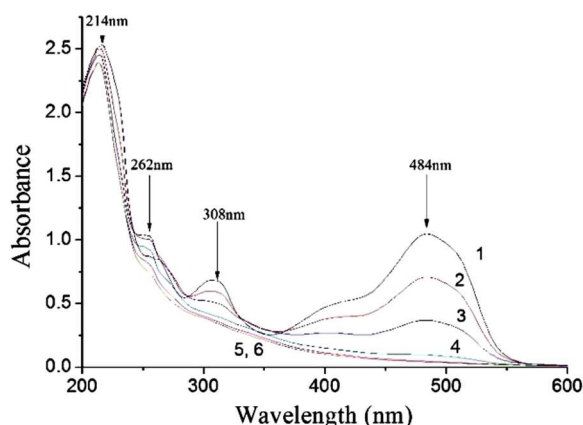
3. Effect of PS concentration on AO7 degradation in Vis/Fe^{3+}/PS process

Different amounts of PS were used to investigate the effect of PS dosage on the degradation of AO7 in Vis/Fe^{3+}/PS process. Fig. 3 illustrates the removal efficiency of AO7 at different PS concentrations with initial AO7 concentration 20 mg/L, Fe^{3+} concentration 0.1 mM, initial pH 3.46 and the light intensity 500 W. When PS concentration increased from 0 to 0.2 g/L, the removal efficiency of AO7 increased from 48% to 95% within 20 min. However, further increase of the PS dosage to 0.4 g/L and 0.8 g/L resulted in only a slight increase in the AO7 removal, and the removal efficiency improved slightly to 97% when PS is 0.8 g/L. This can be explained by the unfavorable consumption of sulfate radicals by the excessive dosage of PS, which has a scavenging potential for sulfate radical (Eqs. (5)).

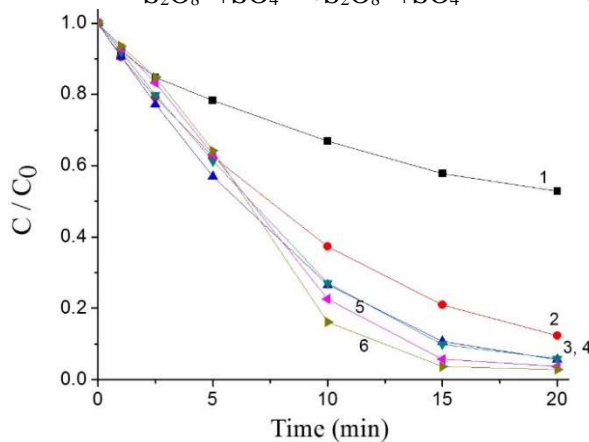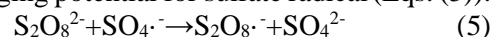$$S_2O_8^{2-}+SO_4\cdot^- \rightarrow S_2O_8\cdot^- +SO_4^{2-} \qquad (5)$$

Fig. 3. The effect of PS concentration (g/L): (1) 0, (2) 0.05, (3) 0.1, (4) 0.2, (5) 0.4, (6) 0.8
Рис. 3. Влияние концентрации персульфата, г/л: (1) 0, (2) 0,05, (3) 0,1, (4) 0,2, (5) 0,4, (6) 0,8

Hence, in order to improve the degradation of probe compounds at the highest extent and reduce the cost, a proper dosage of PS should be selected in the application. Then, keeping the concentration of PS at 0.2 g/L, the effect of various Fe^{3+} dosages on the AO7 degradation was studied in the subsequent experiments.

4. Effect of Fe^{3+} concentration on AO7 degradation in Vis/Fe^{3+}/PS process

The effect of Fe^{3+} concentration on AO7 degradation efficiency was evaluated by conducting experiments at 0.025, 0.05, 0.1, 0.15 and 0.2 mM, respectively. The initial AO7 concentration was fixed at 20 mg/L, PS concentration was 0.2 g/L, initial pH was 3.46 and the light intensity was 500 W. As shown in Fig. 4, when Fe^{3+} concentration varied from 0.025 to 0.1 mM, the removal efficiency of AO7 within 20 min reaction increased from 60.9% to 94.3%. The higher efficiency of AO7 removal at the higher Fe^{3+} dosage might due to more amount of available Fe^{2+} generated and then more radicals produced in the reaction. Further increasing the Fe^{3+} concentration to 0.2 mM resulted in almost the same removal of AO7 with Fe^{3+} 0.1 mM. Therefore, 0.1mM Fe^{3+} is required to produce an essential amount of Fe^{2+} and then effectively activate PS to degrade AO7.
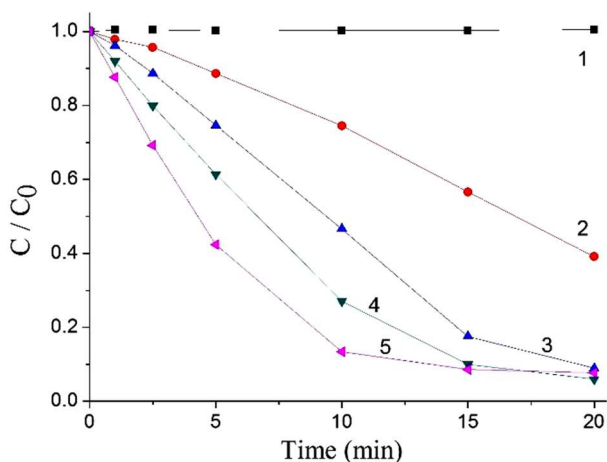

Fig. 4. The effect of Fe^{3+} concentration (mM): (1) 0, (2) 0.025, (3) 0.05, (4) 0.1, (5) 0.2
Рис. 4. Влияние концентрации Fe^{3+}, мМ: (1) 0, (2) 0,025, (3) 0,05, (4) 0,1, (5) 0,2

5. Effect of different initial concentrations of AO7

Fig. 5 shows the changes of AO7 relative concentration as a function of irradiation time at initial AO7 concentrations of 20, 50 and 100 mg/L. The degradation of AO7 has an identical trend of concentration decay as the exposure time increases, while removal percentage of AO7 decreased from 66 to 10% with the increase in initial AO7 concentration from 20 to
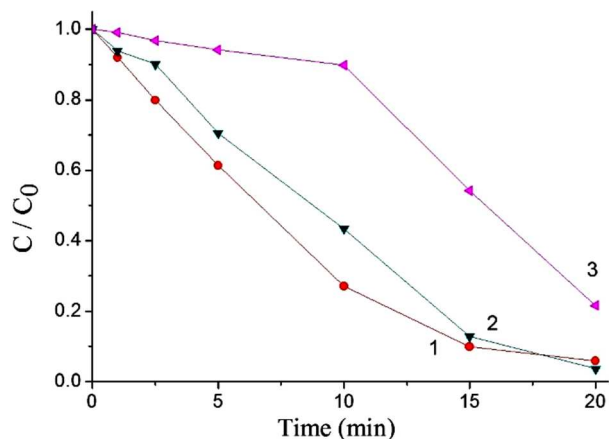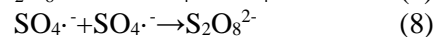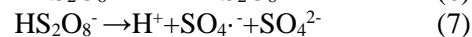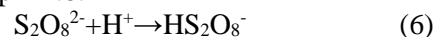

Fig. 5. The effect of AO7 concentration (mg/L): (1) 20, (2) 50, (3) 100
Рис. 5. Влияние концентрации АО7, мг/л: (1) 20, (2) 50, (3) 100

100 mg/L in 10 min, implying that the apparent reaction rate constant with a low initial AO7 concentration is higher than that with a high initial AO7 concentration. This may be explained that the visible light transmittance could be decreased due to an increase of AO7 concentration in the solution, which led to scattering and shielding of the light.

6. Effect of initial pH on AO7 degradation in Vis/Fe^{3+}/PS process

The solution pH can significantly affect degradation of organic pollutants in chemical oxidation processes. The effect of initial pH experiments were conducted over a wide pH range (1.8-9.21). Four levels of pH = 1.8, pH = 3.46, pH = 6.16 and pH = 9.21 were prevailed to evaluate the pH effect on AO7 degradation in Vis/Fe^{3+}/PS system under condition of 0.2 g/L PS, 0.1 mM/L Fe^{3+}, 500 W visible light and 20 min reaction time. As depicted in Fig. 6, the best performance of Vis/Fe^{3+}/PS was observed in pH 3.46 with 94.3% removal efficiency. In acidic condition with high concentration of protons, the generation of sulfate radical was enhanced through acid-catalyzation in accordance with Eqs. (6) and (7). In the higher sulfate radical concentrations, self-scavenging of sulfate radical occurred (Eq. (8)) which had a detrimental effect on AO7 degradation. Therefore, the efficiency of Vis/Fe^{3+}/PS system decreased at pH 1.8.

$$S_2O_8^{2-}+H^+\rightarrow HS_2O_8^- \tag{6}$$
$$HS_2O_8^-\rightarrow H^++SO_4\cdot^-+SO_4^{2-} \tag{7}$$
$$SO_4\cdot^-+SO_4\cdot^-\rightarrow S_2O_8^{2-} \tag{8}$$

On the other hand, with an increase in pH, ferric ion can be precipitated in form of hydroxide contributing to the removal of soluble iron catalyst, which could be the main cause of a negligible change in the degradation efficiency at pH 6.16 and pH 9.21.
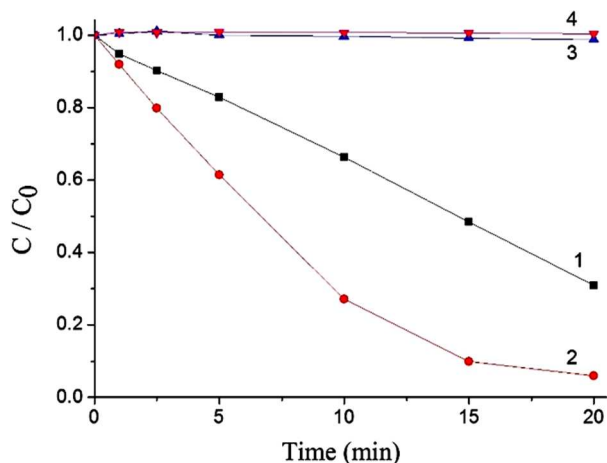
Fig. 6. The effect of pH: (1) 1.8, (2) 3.46, (3) 6.16, (4) 9.21
Рис. 6. Влияние pH: (1) 1,8, (2) 3,46, (3) 6,16, (4) 9,21

7. Effect of co-existing inorganic anions

The presence of common anions in real dye-containing wastewater may affect the efficiency of dye degradation reaction. To investigate the effects of the inorganic anions, such as NO_3^-, SO_4^{2-}, Cl^-, HCO_3^- and HPO_4^{2-}, a series of experiments were conducted, and the results are given in Fig.7. It can obviously be found that Cl^- and NO_3^- slightly affected the degradation of AO7 in Fe^{3+}/PS/Vis system, and SO_4^{2-} had certain influence on the AO7 degradation from 95% to 85% within 20 min. However, the addition of HCO_3^- and HPO_4^{2-} almost completely inhibited the degradation of AO7 in Vis/Fe^{3+}/PS process. Their inhibiting effects can be ranked from low to high in an order of

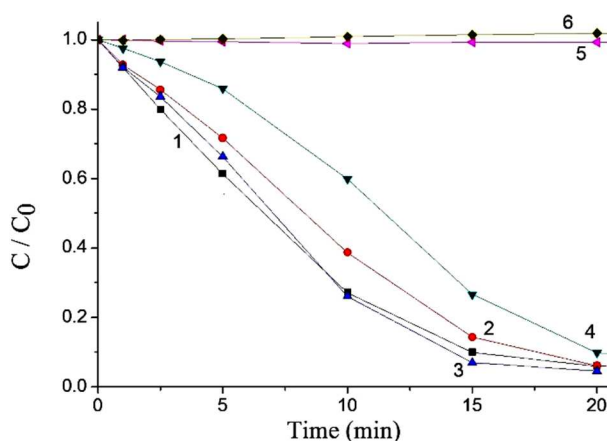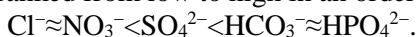$$Cl^- \approx NO_3^- < SO_4^{2-} < HCO_3^- \approx HPO_4^{2-}.$$

Fig. 7. The effect of negative ions on the initial degradation rate:
(1) Control, (2) NO_3^-, (3) Cl^-, (4) SO_4^{2-}, (5) HCO_3^-, (6) HPO_4^{2-}
Рис. 7. Влияние отрицательных ионов на нчальную скорость:
(1) контроль, (2) NO_3^-, (3) Cl^-, (4) SO_4^{2-}, (5) HCO_3^-, (6) HPO_4^{2-}

CONCLUSION

In this study, the degradation of AO7 by Fe^{3+}/PS/Vis system was investigated, and it was demonstrated that Fe^{3+}/PS process under Vis light was an effective method for the degradation of AO7 azo dye in acidic aqueous medium. The effect of AO7 concentration, PS concentration, Fe^{3+} concentration and initial pH, on the AO7 decolorization was investigated. The results indicated that AO7 can be totally decolorizated in a 20 min reaction under the conditions of initial AO7 concentration 20 mg/L, PS concentration 0.2 g/L, Fe^{3+} concentration 0.1 mM/L and Vis light 500 W. The effects of the inorganic anions that likely influence the degradation rate in Fe^{3+}/PS/Vis system were also investigated. HCO_3^- and HPO_4^{2-} had a negtive effect on AO7 degradation efficiency in Fe^{3+}/PS/Vis progress. The data will be helpful for the development of simple and efficient Fenton-like systems aimed to pollutants degradation and, as a result, for designing the energy-resource-saving chemical processes of water management systems of industrial plants [28].

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