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Abstract
Advanced oxidation is one of the most effective processes for degradation of textile industrial swage. The aim of this project was investigation of removal of the (AR14) and (DR23) in presence of dual oxidants H2O2, S2O52- in presence of catalyzers like Fe(II), magnetic iron oxide nanoparticles ions. Fe3O4 composite nanoparticles with reusability and photo catalytic property were synthesized by precipitation method. The morphology, size and structure properties of the resulting Fe3O4 nanoparticles were characterized by SEM and XRD. The advantage of usage of dual oxidants is to producing more effective radicals and high ratio of removal in comparison of one oxidant. The variables that change in this strategy was concentration of two oxidants ,concentration of pollutant (AR14) and (DR23), while content of Catalysts or dosages of them, pH and the constant rate (K) were studied. The decrease of the adsorption of solution in max=514nm and max =507 nm depicts the degradation of the (AR14), (DR23) by passing the time .The result of this study is Catching higher ratio removal in presence of dual oxidants, also beneficial economically and indicate high quality of (AOPs) in wastewater treatment.

Keywords: Advanced Oxidation Processes, Potassium peroxomonosulfate, Direct Red 23, Acid Red 14, water and wastewater treatment.

Introduction
Nowadays, compounds and toxic and resistant materials in water and production of wastewater containing toxic and complex compounds have limited use of conventional processes for water and wastewater treatment. Over the past two decades, environmental standards have become more stringent. However, due to the need to provide public health and environmental protection, attention to efficient technologies is essential in this field. So, in the last fifteen years various treatment technologies have developed from technical and economical perspectives. One of these treatment technologies are well known as Advanced Oxidation Processes. Systems (AOPs) have been taken into account as a powerful and efficient technology and a key technology for the future [Chong, M. N]. One of the chemical properties of advanced oxidation processes is hydroxyl radical production and the high ability of this radical to launch oxidation processes to achieve removal of organic contaminants. Oxidants such as monosulphate proxy and disulfate proxy which generate sulphate radicals(SO4) have been more likely taken into account due to strong oxidation(1.82V) from H2O2(1.76 V). SO4 raises through breaking peroxide bonds by means of radiation, optical and thermal activation of persulfate or electron transfer from the activation of monosulphate proxy and disulfate proxy by the transferor metals. Advanced oxidation methods based on SO4 are used for the decomposition of organic material [Jie.Wu, Chen, X]. Advanced oxidation processes are usually used with a UV beam together with homogeneous or heterogeneous catalysts to reduce pollutions.
Currently, the oxidants such as hydrogen peroxide and sulfate proxy in the presence of UV light and/or metal catalysts such as titanium dioxide and zinc oxide are used [Wang, Y.R]. recent studies have shown that visible light is also capable of degrading organic materials and various dyes [Ding, X]. Light causes excitation of catalyst electrons and causes a hole which this hole can directly react with molecules of organic substances and/or indirectly oxidizes by creation of free radicals of molecules of organic material [Kusvuran, E.]. in recent years, generation of active oxidizing species by electrochemical advanced oxidation methods as an Effective and Environmentally Friendly Method has been largely examined in the context of removal of contaminants and wastewater treatment. These methods are used in removing persistent organic pollutants that cannot be removed biologically during wastewater treatment [Ding, X, Wang, Y].

**Samples of research in removal of acidic 14 different advanced oxidation processes**

Rasouli fard and his colleagues (2011) studied the extent of removal of organic dyes as the coloring material through photo-oxidation process UV/S₂O₈²⁻. They investigated the degradation of Acid Red 14 (AR14), commonly used as a textile dye in aqueous medium through the oxidation process by UV/S₂O₈²⁻ under a set of variables concentration of S₂O₈²⁻, Ag⁺, AR14 and temperature. Commonly Ag⁺, heat and UV light can excite S₂O₈²⁻ to sulfate radical form (SO₄²⁻), a stronger oxidant (E⁰ = 2.60 V) than S₂O₇²⁻, to enhance significantly the oxidation of contaminants. Also the changes in the absorption spectra of AR14 solutions during the photodegradation process showed that decrease of absorption peak of the dye at \( \lambda_{max} = 514 \) nm indicates a rapid degradation of the azo dye. The results of this study suggest that the oxidative treatment of AR14 by peroxydisulfate with UV is a viable option for removal of the textile dyes from effluents (Rasouli fard, MH, 2011). Sadegh pour et al.(2011) examined degradation of acid red 14 through (UV-LED)/S₂O₈²⁻UV. Degradation has not occurred by use of UV light, while 31% of degradation of dye has occurred by use of persulfate and 90% of degradation has occurred by use of persulfate and UV. In this study, a series of effective variables on amount of removal including concentration of S₂O₈²⁻, concentration of dye, radiation pulse, temperature, PH. The reactor designed by LEDs, effect of current on the degradation and the amount of electrical energy per unit (EE0) has been investigated. Comparison of E⁰ between the LED and conventional lamps in different concentrations of persulfate has displayed more efficiency of LEDs. Results from research have displayed that the oxidation process using peroxydisulfate with optimum concentration 120 mM at neutral pH as well as ultraviolet light that is pulsed light 8.0 and off 2.0 has had the highest degree of acid red 14 (AR14)/Sadegh pur, M. 2011. Nazari and colleagues (2012) in a new study examined the efficiency of rectangular photosynthesis in the presence of UV light to remove acid red 14 (AR14). In this research, nanoparticles (TiO₂) as photocatalysts were stabilized by white cement on tiles parts. The factors including weight ratio of TiO₂ to cement, pH and dye concentration have been investigated in this research. Ultimately, natural pH and room temperature have been determined as optimal conditions to remove dye(Nazari, P.). Samples of the research on removal of organic pollutants from contaminated waters via AOPs/S₂O₈²⁻ process. Ding & Zhang(2012) conducted a study entitled “Design of a visible light driven photo-electrochemical/electro-Fenton coupling oxidation system for wastewater treatment” in which they reported on a photo-electrochemical/electro-Fenton oxidation (PEC/EF) system by coupling visible light driven photo-electrochemical oxidation (PEC) and electro-Fenton oxidation (EF) in an undivided cell. Bi₂WO₆ nanoplates deposited on FTO glass (Bi₂WO₆/FTO) and Fe@Fe₃O₄ core–shell nanowires supported on activated carbon fiber (Fe@Fe₃O₄/ACF) were used as the anode and the cathode in the PEC/EF system, respectively. This novel PEC/EF system showed much higher activity than the single PEC and EF systems on degradation of rhodamine B in aqueous solution at natural pH. Moreover, the degradation and the instantaneous current efficiencies of the PEC/EF system were increased by 154% and 26% in comparison with the sum of those of single PEC and EF systems, respectively. These significant enhancements could be attributed to the synergistic effect from better separation of photo-generated carriers in the photo-anode and the transfer of photo-electrons to the oxygen diffusion cathode to generate more electro-generated H₂O₂ and hydroxyl radicals on the Fenton cathode. The better separation of photo-generated carriers contribute more to the overall degradation enhancement than the photo-electrons generated H₂O₂ and the subsequent Fenton reaction on the cathode during the PEC/EF process. Yao-Hui Huang et al.(2009) conducted a study entitled “Efficient decolorization of azo dye Reactive Black B involving aromatic fragment degradation in buffered
Co2+/PMS oxidative processes with a ppb level dosage of Co2+-catalyst” and said that in order to generate powerful radicals as oxidizing species for the complete decolorization and degradation of azo dye Reactive Black B (RBB) at near neutral pH (pH 6), homogeneous activation of peroxymonosulfate (Oxone: PMS) by the trace Co2+-catalysts was explored. We not only took advantage of the high oxidation-reduction potential of produced hydroxyl and sulfite radicals but also an opportunity to oxidize RBB to less complex compounds with extremely low dosages, especially the ppb level of the Co2+-catalyst (stoichiometric ratio: \([\text{Co}^{2+}]/[\text{RBB}](0)=1.7 \times 10^{-6}-1.7 \times 10^{-5} \); \([\text{PMS}](0)/[\text{RBB}](0)=8 \times 32\)). Anion effects and pH effects were also carried out and discussed to simulate an actual application such as that of a textile waste stream. Both the degradations of RBB and its derivative aromatic fragments were illustrated successfully at UV-visible absorptions of 591 and 310 nm, respectively, and the possible relationships between them were also proposed and discussed, based on the experimental results. The RBB degradation in this Co2+/PMS oxidative process successfully formulated a pseudo-first-order kinetic model at an isothermal condition of 25 degrees C with or without different anions present. The initial rate and rate constant were calculated under different comparative conditions, and the results indicate that the activity of both RBB decolorization and its degradation are not obviously dependent on the PMS concentration, but rather are related to the Co2+ dosage (Yao-Hui, H).

Materials and methods

Materials
1. Acid Red 14 (99 % Alvan Sabet Loom Stuff & Paint Production Co, Tehran)
2. Peroxymonosulfuric acid (Sigma)
3. Iron (II) sulfate (Merck)
4. Sulfuric acid (Merck)
5. Sodium hydroxide (Merck)
6. Twice distilled water distillation apparatus (GFL, type: 2008 / Germany)

Preparation of organic dye Acid Red 14 stock solution
To prepare the organic dye from aqueous solution with concentration 1000 mg/l, amount of 0.1 grams of it has been dissolved in distilled water and delivered in a 100 ml balloon in volume, which organic dye is used to provide the desired concentration.

Preparation of a solution of potassium peroxymonosulfate
To prepare the potassium peroxymonosulfate solution with a concentration of 10 mM, 0.3078 grams of this material are dissolved with 99% purity in distilled water and brought to a volume of 100 ml, used to prepare the considered concentrations.

Preparation of soluble iron (II) sulfate
To prepare soluble iron (II) sulfate with a concentration of 10 mM, 0.2780 grams of this material are dissolved in distilled water and brought to a volume of 100 ml, used to prepare the considered concentrations.

Providing a solution of sodium hydroxide and sulfuric acid (to adjust pH)
According to the desired concentrations, a certain weight of solid sodium hydroxide and a certain volume of sulfuric acid have been taken and brought to a certain volume with distilled water.

The method

Measuring the concentration of dye Acid Red 14
Spectrophotometry refers to one of the methods to measure the concentration of dye pollutants, so that spectrophotometry UV-Vis is one of these methods. To obtain the maximum wavelength of dye Acid Red 14, the solutions with certain concentration were prepared and their spectrum was obtained to display their stability at various PHs. dyes Acid Red has been displayed in figure 1 and displayed at various PHs in figure 2. With regard to figure 2, it can observe that the maximum wavelength of dye Acid Red 14 at various PHs has not been displayed with tangible change, thus it can assume it constant at all PHs equal to \(\lambda_{\text{max}}=514\) nm.
Discontinuous measurement method of dye materials
With regard to the experiments under study for the purpose of preparation of certain concentrations of oxidants, a certain volume of the soluble dye material and peroxymonosulfate brought to the final volume of 100 ml with distilled water.

These calculations follow the rule follow:

\[ \sum \text{ml} \cdot 100 \text{volume of distilled water} (\text{+ dye volume and peroxydisulfate volume}) \]

Then, with regard to type of parameter under study, conditions of solution such as concentration of dye material and concentration of oxidant are regulated. In each of the prepared solutions, initial uptake of solution is measured before using any of the oxidizing agents of peroxymonosulfate such as light and metal catalyst. After starting experiment, sample is taken from the solution under study and the sample uptake is measured via spectrophotometer UV-Vis during experiment (10 min). Thus, during experiment amount of uptake of dye in the solution has been measured and charts (X) against time (t) related to it is drawn. (X) is the ratio of the concentration of the dye material at the moment (t) after beginning reaction to the initial concentration of dye material . If \( C_0 \) represents initial concentration of dye and \( C_t \) represents concentration of dye at moment (t), then

\[ X = \frac{C_t}{C_0} \quad 0 \leq X \leq 1 \]

If \( X=1 \), there will be \( C_t=C_0 \), i.e. lack of change in initial concentration implies that nothing has removed until t moment; if \( X=0 \), there will be \( C_t=0 \), i.e. the dye has removed and the concentration has reached to 0. With regard to Beer-Lambert law, amount of dye uptake (A) and dye concentration (C) are consistent with each other.
A \propto C \Rightarrow A = \varepsilon b C

Then it can obtain X from the equation below and introduce it as the ratio of solution uptake at moment t after beginning reaction to the initial solution uptake:

\[ X = \frac{A_I}{A_0} \]

Here, with regard to Beer-Lambert law and equation 4, it can obtain percent of dye removal at moment t after starting reaction:

\[ \% \text{ dye removal} = \frac{A_0 - A_t}{A_0} \times 100 \]

To calculate amount of dye material removal at various periods of time, beginning oxidation processes related to equation 4 is used.

**The calibration curve**

To make the relationship between amount of measured uptake and amount of red acid dye 14 concentration which is changing during the removal process, it requires using a chart. In this regards, it can relate the changes in solution uptake at maximum uptake wavelength to the changes in concentration. For this, the solutions with various concentrations at linear range of Beer-Lambert law were prepared and the uptake of these solutions was measured. The solutions were prepared and uptake of red acid dye solutions were measured with high accuracy, because the curve obtained between uptake and concentration of dye solution will be used in next parts.

![Figure 3. Calibration chart of red acid dye 14 solution at wavelength 514 nm](image)

**Design of Experiments to determine the optimal concentration of peroxymonosulfate (first oxidant)**

To determine optimal concentration of peroxymonosulfate, the samples with 0.1 mM concentration of Iron ions(Fe$^{2+}$) and 20mg/l of dye material and different concentrations of peroxymonosulfate are prepared at natural PH of dye solution. Any experiment was made at period of time (60 min) and concentration of peroxymonosulfate in solution was changed during each experiment. With regard to the change of peroxymonosulfate concentration, 9 experiments with concentrations (0, 0.1, 0.5, 1.5, 2, 3, 4, 5 mM) were made at period of time (60 min). During each experiment, firstly initial solution uptake ($A_0$) before adding oxidant is read and then sample is taken from the solution after adding oxidant during period of time (10 min) and solution uptake ($A_t$) is measured via spectrophotometer device. Thus, after 60 minutes from beginning experiment, the number related to uptake represents the amount of dye removal via device. Ultimately, dye removal ratio($X$) against reaction time ($t$(min)) has been drawn, then optimal peroxymonosulfate oxidant amount is determined and considered constant to examine next parameters.
Design of experiments on iron ions
To determine optimal concentration of catalyst, the solutions at ultimate volume (100 ml) with constant dye concentration (20 mg/l) and optimal amount of monosulfate oxidant concentration (1mM) were obtained and the concentrations 0.2, 0.5, 0.075, 0.1, 1.5, 2, 2.5 and 5 mM of iron ion were prepared at environment temperature and natural PH. Concerning each of solutions before adding oxidant, firstly initial uptake of solution containing dye contaminant and iron ion was measured and then the experiment starts by adding two oxidants to the solution and ultimately solution uptake was measured per 10 minutes.

Design of experiments on effect of dye concentration on how the system works out
To examine effect of dye concentration, the solutions at volume 100 ml with 1 mM peroxymonosulfate concentration and iron catalyst, red acid dye 14 with concentrations 5, 20, 10, 30, 40 and 50 mg/l at natural PH of solution and environment temperature at volume 100 ml were prepared. Firstly the initial uptake (A₀) of solution was measured and then the sample was taken from the solution during 60 minutes at period of time (10 min) and ultimately uptake amount of solution (Aₜ) was measured. Ultimately, effect of dye concentration amount on removal amount was examined by drawing the removal amount against time.

Design the experiment on effect of PH
In this section, firstly the solutions with initial dye concentration 20 mg/l and 1mM peroxymonosulfate and iron ion catalyst and distilled water to bring to final volume of 100 ml were prepared. Then, initial pH of solutions was adjusted by adding the required quantities of sulfuric acid or sodium hydroxide depending on the desired pH. For this, after adding distilled water, dye and iron ion to PH solution were adjusted and then volume of mentioned oxidants was poured to the solution and then the oxidation reaction and dye material removal were started by entering oxidants to solution. PHs considered include 2, 3, 4, 5, 7, 9 and 11. Effect of PH on amount of dye removal can be examined by taking sample from solutions and measuring amount of uptake of samples via previous method and drawing the charge of change in uptake amount against time changes.

Experiment on effect of temperature
To determine effect of temperature, the solutions at ultimate volume (100 ml) with constant dye concentration (20 mg/l) and optimal amount of monosulfate oxidant concentration (1mM) were obtained and the 10, 20, 25, 30, 40, 50,60 and iron ion were prepared at environment temperature and natural PH. Firstly initial uptake (A₀) of solution was measured and then the sample was taken from the solution and the uptake amount (Aₜ) was measured and ultimately effect of temperature on removal amount was examined by drawing removal amount against time.

Study on Reaction Kinetics
Degradation kinetics study by taking sample from experiment solutions was made at certain period of time.

The results from study on parameters affecting removal of red acid dye 14 using $\text{S}_2\text{O}_5^2-/\text{Fe}^{2+}$
In study on Catalytic oxidation of red acid dye 14 against parameters affecting $\text{S}_2\text{O}_5^2-/\text{Fe}^{2+}$, data from pre-designed experiments were drawn and analyzed in the associated diagrams.

Study on effect of Concentration of antioxidants/catalyst and their comparison
Changes in red acid dye 14 uptake in terms of time for the experiments in the presence of single $\text{Fe}^{2+}$, single $\text{S}_2\text{O}_5^2-$ and $\text{S}_2\text{O}_5^2-/\text{Fe}^{2+}$ have been displayed in figure 4. As observed, there is no substantial change in amount of red acid dye 14 removal in the absence of oxidant and presence of single $\text{S}_2\text{O}_5^2-$. Iron ions with activation of peroxymonosulfate and hydrogen peroxide and creation of sulphat radicals have caused dye removal.
Figure 4. study on effect of single Fe$^{2+}$ and single S$_2$O$_5^{2-}$ in comparison with S$_2$O$_5^{2-}$/Fe$^{2+}$ on red acid dye 14 removal

$[S_2O_5^{2-}]_0 = 1\text{mM}, [Fe^{2+}]_0 = 0.1\text{mM}, \ pH_0 = \text{natural} \ [AR14]_0 = 20 \text{ppm}$

Results and analysis of experiments on effect of initial concentration of potassium peroxymonosulfate

The results from study on monosulfate concentration (0-5 mM) on dye removal process have been displayed in figure 5. With regard to figure, it is observed that there is a direct relationship between initial monosulfate concentration and amount of dye removal, that is, the amount of dye removal increases by increasing initial monosulfate concentration and the sulphate radicals increase by increasing monosulfate concentration; further generated hydroxyl radicals assist for further dye degradation.

$S_2O_5^{2-} \rightarrow 2 SO_4^{2-}$

$SO_4^{2-} + H_2O \rightarrow HO^\cdot + H^+ + SO_4^{2-}$

Removal (61.92%) was obtained at 1 mM concentration of monosulfate during 60 minutes which selection of this amount is suitable, not required for further removal percent. At higher concentrations of mentioned concentration, ascending trend of dye material removal diminishes. This phenomenon can be explained in this way that concentration of free and active sulfate radicals and/or generated hydroxyl increase by increasing initial concentration of peroxymonosulfate that the particles collide with each other and combine with each other before they collide to dye material molecules and cause degradation of these materials.
According to equations 7 and 8, sulfate radicals generate $\text{S}_2\text{O}_5^{2-}$ in collision with each other and hydroxyl radicals generate $\text{H}_2\text{O}_2$ in collision with each other. By increasing concentration of hydrogen peroxide, hydrogen peroxide works out with hydroxyl radical probing, under which the efficiency of system does not increase under this interaction as expected.

$2\text{SO}_3^{-} \rightarrow \text{S}_2\text{O}_5^{2-}$
$2\text{OH}^{-} \rightarrow \text{H}_2\text{O}_2$ (acidic and)
$\text{OH}^{-} + \text{H}_2\text{O} \rightarrow \text{H}_2\text{O} + \text{OH}^{-}$
$\text{OH}^{-} + \text{SO}_4^{*} \rightarrow \text{HSO}_4$

Results and analysis of experiments to determine the optimal concentration of iron ion catalyst in $\text{Fe}^{2+}/\text{S}_2\text{O}_5^{2-}$

With regard to figure 6, results from study on effect of concentration of iron ion catalyst in $\text{Fe}^{2+}$ on amount of dye removal indicates that amount of dye removal increases by increasing $\text{Fe}^{2+}$ concentration in solution, because increasing iron ion concentration as the activator of peroxymonosulfate causes proliferation of peroxymonosulfate to sulfate radical which is a stronger oxidant, thus amount of dye material degradation increases through generated sulfate radicals.

Results and analysis of experiments on effect of initial concentration of dye material in $\text{Fe}^{2+}/\text{S}_2\text{O}_5^{2-}$

Study on effect of initial concentration of pollutant on removal amount refers to one of the important parameters in amount of efficiency of advanced oxidation systems. Results from study on this effect in efficiency of $\text{Fe}^{2+}/\text{S}_2\text{O}_5^{2-}$ have been displayed in figure 7.
With regard to figure, it is observed that dye removal amount decreases by increasing initial concentration of red acid dye 14 from 5 ppm to 50 ppm under general state of fixed concentration of peroxymonosulfate and iron ion catalyst. It can justify this in such a way that since concentration of peroxymonosulfate and iron ion catalyst is fixed, increase in dye concentration against fixed concentration of generated sulfate radicals has caused decrease in amount of dye removal.

**Study on changes in Absorption spectra before and after removal of red acid dye 14**

In figure 8, Absorption spectra of red acid dye 14 at range of 200 to 800 nm at various periods of time has been displayed per 10 minutes since starting oxidative degradation reaction. Maximum peak of red acid dye 14 is at 514 nm wavelength. With regard to figure, it can observe that the peak intensity at absorption spectra of dye material decreases by progressing the reaction and the amount of absorption at these absorption peaks declines, indicating degradation and removal of dye molecules.

**Results and analysis of experiments on effect of initial PH in efficiency of system**

Effect of PH changes on amount of dye removal depends on type and conditions of system and dye material. At acidic PHs, more sulfate radicals are generated having more efficiency than basic PHs. Further, it has been proven that the highest return occurs at PHs under 3. As observed, removal at PH=4 is greater than PH=2 which is more acidic. The reason lies on this fact that concentration of sulfate radicals activated via acid and sulfate radicals activated via iron catalyst is to a large extent at PH=2 that it can result in radical-radical interaction whereby the interaction between radicals and organic compounds decreases, deducing that amount of dye removal has reduced. At basic environments, hydroxyl radicals increase. Hydroxyl radicals have the same oxidation power to the sulfate radicals. At PH above 10.7, all the sulfate radicals convert to hydroxyl radicals.

\[
\text{SO}_4^{2-} + \text{HO}^- \rightarrow \text{SO}_4^{2-} + \text{HO}^-
\]

![Absorption spectra](image-url)
Results and analysis of experiments on effect of initial temperature on efficiency of system

The results from study on effect of various temperatures in certain concentration of peroxymonosulfate and dye have been displayed in figure 10. As observed, amount of dye removal increases by increasing temperature; Increasing temperature regarding relation $k = k_0 \exp \left( -\frac{E_a}{RT} \right)$ by decreasing activation energy and conversion of oxidant peroxymonosulfate ions to sulfate radicals with further oxidation power will cause more effective removal. Further heat causes stimulation of $S_2O_5^{2-}$ and generation of further radicals and better dye removal. According to $k = k_0 \exp \left( -\frac{E_a}{RT} \right)$, $k_0$ represents collision coefficient, $E_a$ represents activation energy, $R$ represents constant and $T$ represents temperature in terms of Kelvin.

Conclusion

In the present research, removal of red acid dye 14 by means of peroxymonosulfate and hydrogen proxide oxidants which are strong oxidants was made in the absence of visible light and ultraviolet radiation, so that percent of removal of organic dye material is over 90%. Further, cheap and available catalyst at low scale and non-toxic catalyst in this method such as iron sulfate causes less pollution in the environment. In these experiments, concentration of peroxymonosulfate and hydrogen proxide oxidants equal to 1 mM and 0.3 mM and concentration of iron catalyst 1 g.1⁻¹ was examined, indicating a favorable removal to 95%. The kinetic of process follows first order equation.
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