Special issue:
Sustainable water management for green infrastructure

Lee-Hyung Kim (Chief of Editors), Zuwhan Yun, Joohyon Kang, Sungpyo Kim, Yingxia Li, Marla Maniquiz-Redillas

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## Special Issue: Sustainable water management for green infrastructure

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Ultrasound enhanced heterogeneous activation of peroxydisulfate by bimetallic Fe-Co/GAC catalyst for the degradation of Acid Orange 7 in water

Chun Cai, Liguo Wang, Hong Gao, Liwei Hou, Hui Zhang

Department of Environmental Engineering, Hubei Biomass-Resource Chemistry and Environmental Biotechnology Key Laboratory, Wuhan University, Wuhan 430079, China. E-mail: caichun415@163.com

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ABSTRACT
Bimetallic Fe-Co/GAC (granular activated carbon) was prepared and used as heterogeneous catalyst in the ultrasound enhanced heterogeneous activation of peroxydisulfate (PS, S₂O₅²⁻) process. The effect of initial pH, PS concentration, catalyst addition and stirring rate on the decolorization of Acid Orange 7 (AO7) was investigated. The results showed that the decolorization efficiency increased with an increase in PS concentration from 0.3 to 0.5 g/L and an increase in catalyst amount from 0.5 to 0.8 g/L. But further increase in PS concentration and catalyst addition would result in an unpronounced increase in decolorization efficiency. In the range of 300 to 900 r/min, stirring rate had little effect on AO7 decolorization. The catalyst stability was evaluated by measuring decolorization efficiency for four successive cycles.

Nowadays, sulfate radical-based advanced oxidation processes have proven to be the very promising techniques for the treatment of refractory pollutants (Anipsitakis and Dionysiou, 2003; Chan and Chu, 2009; Wang et al., 2011). As a source of sulfate radicals (SO₄⁻²; \( E^{\text{0}} = 2.6 \) V vs. normal hydrogen electrode (NHE)), peroxydisulfate (PS, \( S₂O₅²⁻ \)) is a strong and stable oxidizing agent (\( E^{\text{0}} = 2.01 \) V vs. NHE) and it has high aqueous solubility and high stability at room temperature as compared to hydrogen peroxide (\( H₂O₂; E^{\text{0}} = 1.77 \) V vs. NHE) (Lin et al., 2013). PS can be activated by heat (Waldemer et al., 2007; Xie et al., 2012), microwave (Yang et al., 2009a), UV (Lau et al., 2007; Salari et al., 2009), or transition metals (Anipsitakis and Dionysiou, 2004; Gayathri et al., 2010; Long et al., 2014), shown in Eqs. (1) and (2), thus generating SO₄⁻²⁻ which could oxidize azo dyes into small molecules and carbon dioxide.

\[
S₂O₅²⁻ + \text{heat/UV} \rightarrow 2SO₄⁻²⁻ \quad (1)
\]

\[
S₂O₅²⁻ + \text{Me}^{n+} \rightarrow SO₄⁻²⁻ + SO₄²⁻ + \text{Me}^{(n+1)+} \quad (2)
\]
In the past few years, most studies have focused on single Fe-based and Co-based catalysts for chemical activation of PS (Gayathri et al., 2010; Hou et al., 2012). However, the reaction could not perform effectively at neutral and alkaline solution, which limited the application of Fe catalyst (Zhao et al., 2010; Zhu et al., 2013). Among various transition metal catalysts, Co was considered as a promising candidate due to the reason that Co-containing catalysts possess excellent catalytic performances in a wide range of oxidation reactions at both acidic and alkaline solutions (Anipsitakis and Dionysiou, 2003; Liu et al., 2013; Szegedi et al., 2009). However, the leaching of Co would lead to great threats to our life and environment as a result of their biological toxicity (Shi et al., 2012). Therefore, Fe and Co bimetallic catalyst were introduced to overcome the drawback of Fe and prevent the leaching of cobalt, and thus to improve the utilization of Fe and Co (Su et al., 2012; Yang et al., 2009b). In addition, versatile catalyst supports were utilized to suppress metal leaching (Shi et al., 2012). It is well known that granular activated carbon (GAC) is a good adsorbent and catalyst support. Combination of adsorptive property of GAC and supported metal(s) as a catalyst will provide a good way for aqueous reaction (Shukla et al., 2010). However, few studies have investigated the bimetallic Fe and Co supported on GAC for the activation of PS to degrade the reluctant pollutants.

Recently, more and more attention has been paid to the use of ultrasound irradiation (US) in the treatment of dye wastewater (Chen and Su, 2012; Zhang et al., 2009; Zhong et al., 2011). It was found that ultrasound could act as an activator of \( S_2O_8^{2-} \), as can be seen in Eq. (3) (Price and Clifton, 1996). During ultrasonic irradiation, the collapse of cavitation bubbles induces localized high temperatures and pressures, which allows the formation of oxidizing species, such as ‘OH, superoxide radical (O\(^{2-}\)), as well as the homolytic splitting of polluted compounds (Chen and Su, 2012; Hou et al., 2012). Therefore, the combination of ultrasound with heterogeneous oxidation reaction is a favourable process in the degradation of reluctant organic compounds.

\[
S_2O_8^{2-} + US \rightarrow 2SO_4^- 
\]  

Hence, in this study, Acid Orange 7 (AO7) was chosen as a target azo dye compound and degraded by US enhanced heterogeneous activation process using Fe-Co/GAC as catalyst. The present work aimed at providing insight into the effects of operating parameters, such as initial pH, PS concentration, and stirring rate on the decolorization of AO7. The activity and stability of bimetallic Fe-Co/GAC catalyst during the coupled US/heterogeneous activation process were also explored.

### 1 Materials and methods

#### 1.1 Materials

AO7 (4-(2-hydroxynaphthylazo) benzenesulfonic acid sodium salt) was purchased from Shanghai No. 3 Reagent Factory (China) and used without further purification. The GAC was purchased from Tianjin Bodi Chemical Co., Ltd. (China). All other reagents were of analytical grade and all sample solutions were prepared using deionized water.

#### 1.2 Procedure

Fe and Co oxides were deposited on the as-received GAC (after washing with acid and alkaline solution) by a wet impregnation technique. A fixed amount of GAC was immersed into mixed iron nitrate and cobalt nitrate solution of appropriate concentration, and the suspension was stirred for 24 hr at constant temperature. Then the sample was dried at 120°C for 12 hr and calcined for 4–6 hr under N\(_2\) flow. The metal content was 16 wt%, and the molar ratio of Fe:Co was 2:1. The N\(_2\) Brunauer-Emmett-Teller (BET) method was performed on an ASAP2020 analyzer (USA). The BET surface area, pore size and pore volume of the catalyst were 207.2 m\(^2\)/g, 2.7 nm, and 0.1 cm\(^3\)/g, respectively.

The experimental device used for the study is similar to our earlier research (Hou et al., 2012). A stock solution of AO7 was prepared fresh with deionized water before each run and the initial concentration was fixed at 50 mg/L. Unless otherwise specified, the initial pH of the dye solution was adjusted using 0.1 mol/L sulfuric acid and 0.1 mol/L sodium hydroxide. Then catalyst and PS were added into 200 mL of AO7 solution with a mechanical stirrer (RW 20, IKA, Germany) providing constant stirring around 500 r/min except for the experiments concerning stirring speed. Sonication was performed with a KQ-100 KDB ultrasonic generator (100 W, 20 kHz, Kunshan Ultrasonic Instrument Co., Ltd., China). The reactor was immersed into the ultrasonic batch. All experiments were at constant temperature (22 ± 2°C) by using cooling water through the water circulation system. At selected time intervals, 1.5 mL aliquot was sampled filtered through 0.22 μm membranes (Millipore Co.) and immediately mixed with methanol (AO7 sample:methanol = 1:1, V/V) to quench the reaction before analysis.

#### 1.3 Analysis methods

The absorbance of AO7 was measured at \( \lambda_{\text{max}} = 485 \) nm using a Rayleigh UV-9100 spectrophotometer (Rayleigh Co., China). UV-Vis spectra from 200 to 800 nm were recorded with a UV-1700 spectrophotometer (Shimadzu). The leaching concentrations of Fe and Co were determined by an atomic absorption spectrophotometer (AAS, PerkinElmer, A Analyst 800). The TOC was measured by
a Jena multi N/C 3100 analyze with the non dispersive infrared combustion absorption detector. Cerium catalysts were used in the combustion reaction. Oxygen was provided as a carrier gas, and the flow rate was 150 mL/min. Calibration of the analyzer was obtained by potassium hydrogen phthalate and sodium hydrogen carbonate standards for total carbon (TC) and inorganic carbon (IC), respectively. The TOC value is the difference between TC and IC values.

2 Results and discussion

2.1 Decolorization of AO7 under different systems

Generally, two operating modes are employed to conduct the heterogeneous oxidation. The oxidant is applied in the dye solution simultaneously with the catalyst or after the catalyst is mixed with the solution to reach the adsorption equilibrium. To compare the decolorization efficiency under the two operating modes, a preliminary experiment was carried out. In the presence of US, 98.3% of decolorization efficiency was achieved when PS was dosed together with Fe-Co/GAC catalyst (Fig. 1). However, the decolorization efficiency decreased to 77.7% if the catalyst alone was in contact with the solution beforehand for 30 min. This was possibly attributed to the adsorption of AO7 onto GAC surface. That is to say, the loading dye molecules on the GAC surface decreased the active surface sites available for the activation of PS, which inhibited the degradation of AO7. The result was in consistent with the research by Yang et al. (2011). Consequently, all further experiments were carried out by adding both PS and catalyst at the same time.

As presented in Fig. 1, AO7 was hardly degraded under US alone or PS alone. It may due to the reason that only a very small amount of ‘OH is formed by the ultrasonic fragmentation of water alone (Price and Clifton, 1996). As PS is stable and the chemical reaction rate is relatively slow at room temperature, the decolorization efficiency of AO7 by PS alone was almost the same as that by ultrasonic irradiation alone. When PS was combined with ultrasound, the decolorization efficiency of AO7 increased to some extent (9.4%), indicating PS can be activated by ultrasound but the activation is not efficient to achieve a high color removal. The adsorption ability of Fe-Co/GAC was confirmed because 40.9% of AO7 was adsorbed by Fe-Co/GAC. The further introduction of ultrasound led to a 52.9% decolorization efficiency, showing that only a small amount of active radicals could be produced during the catalytic ultrasonic process in the absence of oxidant. When Fe-Co/GAC catalyst was added to the dye solution with PS, the decolorization efficiency reached 68.9%, clearly illustrating that PS could be activated by Fe-Co/GAC. When ultrasound was introduced into the heterogeneous activated system, 98.3% decolorization efficiency was obtained. The result may be explained by the following reasons. Firstly, the adsorption effect of Fe-Co/GAC accounts for color removal. Moreover, PS could be activated by Fe-Co/GAC to generate SO₄²⁻ at ambient temperature (Eq. (2)). In addition, ultrasound can enhance the decomposition of PS to generate SO₄²⁻ (Eq. (3)).

In order to investigate the catalytic activity of Fe-Co/GAC catalyst, GAC support and Fe-Co/GAC catalyst were used and compared for the decolorization of AO7. The decolorization efficiencies by GAC, US/GAC, GAC/PS, US/GAC/PS were 41.2%, 43.2%, 45.7%, and 47.2%, respectively. However, the corresponding decolorization efficiencies were 40.9%, 52.9%, 68.9%, and 98.3% with Fe-Co/GAC replacing GAC. The results indicated that the catalytic activity of Fe-Co/GAC was much greater than GAC itself. It is due to the reason that PS can react with Fe(II) and Co(II) to generate active sulfate radicals in the following Eqs. (4) and (5) (Gayathri et al., 2010; Hou et al., 2012).

\[
\begin{align*}
S_{2}O_{8}^{2-} + \equiv Fe(II) & \rightarrow SO_{4}^{2-} + SO_{3}^{2-} + \equiv Fe(III) \quad (4) \\
S_{2}O_{8}^{2-} + \equiv Co(II) & \rightarrow SO_{4}^{2-} + SO_{3}^{2-} + \equiv Co(III) \quad (5)
\end{align*}
\]

2.2 Stability of bimetallic catalyst during the US/heterogeneous activation process

The sequential experiments were performed to test the stability of the catalyst. The catalyst was removed from the reactor at the end of each repetitive cycle, then washed by distilled water, dried in the vacuum oven and stored at ambient temperature. As can be seen in Fig. 2, in the primary experiment, AO7 was decomposed 100% after 60 min. The amounts of leached iron and cobalt were determined after 60 min reaction, and the leaching percentages of Fe and Co were 0.76% and 6.28%, respectively. The same amounts of iron and cobalt ions replacing Fe-Co/GAC catalyst were added in the combined US/homogeneous activation process.
process, which achieved 20% AO7 removal. This means the contribution of US/homogeneous activation process due to the leached Co$^{2+}$ and Fe$^{2+}$ to the decolorization of AO7 was not significant. The decolorization efficiency was still up to 96.6% in the fourth cycle, indicating Fe-Co/GAC catalyst is stable and can be reused.

### 2.3 Effect of initial pH on the decolorization of AO7

In order to investigate the influence of initial pH on the degradation of AO7, experiments were carried out at pH 3.0, 5.8, 6.0 and 9.0. The decolorization efficiency only decreased from 99.4% to 97.2% as the initial pH value increased from 3.0 to 9.0 (Fig. 3a). When sulfate radicals were generated via Eqs. (4) and (5), it could react with H$_2$O or OH$^-$ to produce hydroxyl radicals (Eqs. (6) and (7)) (Liang et al., 2007; Hou et al., 2012).

\[
\text{SO}_4^{2-} + \text{H}_2\text{O} \rightarrow \text{SO}_4^{2-} + \cdot \text{OH} + \text{H}^+ \tag{6}
\]

\[
\text{SO}_4^{2-} + \text{OH}^- \rightarrow \text{SO}_4^{2-} + \cdot \text{OH} \tag{7}
\]

Based on Eqs. (6) and (7), H$^+$ would be produced and OH$^-$ would be consumed, which resulted in a reduction in the solution pH. Moreover, H$^+$ would be further released by the dissociation of HSO$_4^-$, which was formed through the reaction of hydroxyl radical and sulfate radical (Eq. (8)) as well as the decomposition of PS in water (Eq. (9)) (Wu et al., 2012).

\[
\text{SO}_4^{2-} + \cdot \text{OH} \rightarrow \text{HSO}_4^- + 1/2\text{O}_2 \tag{8}
\]

\[
\text{S}_2\text{O}_8^{2-} + \text{H}_2\text{O} \rightarrow 2\text{HSO}_4^- + 1/2\text{O}_2 \tag{9}
\]

\[
\text{HSO}_4^- \rightarrow \text{SO}_4^{2-} + \text{H}^+ \tag{10}
\]

Therefore, pH would decrease to almost the same value and the decolorization of AO7 tended to be conducted under the similar conditions as the reaction proceeded. Then the similar decolorization efficiency was observed at different initial pH values.

To further investigate the effect of pH, experiments were conducted with buffer solution (H$_2$SO$_4$–KH$_2$PO$_4$ solution to reach pH 3.0 and NaOH–K$_2$HPO$_4$ solution to pH 9.0). As shown in Fig. 3b, the decolorization efficiencies at pH 3.0 and 9.0 were 98.4% and 91.2% in the 1st cycle, respectively. It indicated that high removal efficiency of AO7 could be obtained under acidic and alkaline solution though the decolorization rate at pH 3.0 was faster than at pH 9.0. In the meanwhile, the stability of the catalyst was tested by performing repeated batch experiments. The decolorization efficiency in the 3rd cycle was 91.9% at pH 3.0, while it decreased to 81.6% at pH 9.0. Since the alkaline activation of PS is insignificant at pH 9 (Chan et al., 2010; Criquet and Leitner, 2009), the results justified Fe-Co/GAC catalyst was stable and active under acidic as well as alkaline conditions.

Based on the above results, the natural pH of AO7 (pH 5.8) without buffer was chosen for subsequent experiments.

### 2.4 Effect of PS concentration on the decolorization of AO7

The decolorization efficiency of AO7 increased significantly when the PS concentration increased from 0.3 to
0.5 g/L (Fig. 4). However, the decolorization efficiency of AO7 could not be improved with the further increase in PS concentration up to 1.0 g/L. An increased amount of sulfate radicals would be generated at a higher PS concentration, but the excessive PS would scavenge sulfate radical as shown in Eq. (11) (Waldemer et al., 2007; Lin et al., 2013). Therefore, 0.5 g/L of PS was selected in the following experiments.

\[
\text{SO}_4^{2-} + \text{SO}_4^{2-} \rightarrow \text{SO}_5^{4-} + \text{SO}_4^{2-} \quad (11)
\]

### 2.5 Effect of catalyst dosage on the decolorization of AO7

The decolorization of AO7 at different Fe-Co/GAC dosages was investigated. As presented in Fig. 5, the decolorization efficiency increased with increasing Fe-Co/GAC dosage from 0.5 to 0.8 g/L. Fe-Co/GAC is a multivalent catalyst, which acts as a good provider of \( \equiv \text{Fe}(II) \) and \( \equiv \text{Co}(II) \) for the activation of PS to generate \( \text{SO}_4^{2-} \). Therefore, the higher loading of Fe-Co/GAC corresponded to the higher decolorization rate of AO7. However, further increase in catalyst amount beyond 0.8 g/L led to an insignificant increase in the decolorization efficiency though the adsorptive removal of AO7 by Fe-Co/GAC increased continuously with catalyst loading from 0.5 to 1.0 g/L. It indicated that the amount of \( \text{SO}_4^{2-} \) generated was sufficient enough to oxidize AO7 when 0.8 g/L or more of Fe-Co/GAC was applied to activate PS.

### 2.6 Effect of stirring rate on the decolorization of AO7

It is regarded that stirring may perturb the formation of cavitation bubbles (Selli et al., 2005; Theron et al., 1999). In order to evaluate the effect of stirring on the decolorization of AO7, experiments with different stirring rates were performed. As can be seen in Fig. 6, the decolorization efficiency was nearly the same when the stirring rate was in the range of 300 to 900 r/min. It illustrated that stirring had little effect on the decolorization of AO7 under the US/heterogeneous activation system.

### 2.7 UV-Vis spectra changes and mineralization of AO7

To clarify the changes of molecular and structural characteristics of AO7 in the US/heterogeneous activation process, representative UV-Vis spectra changes in the solution as a function of reaction time were observed. It was shown in Fig. 7a that absorption spectrum of AO7 in aqueous solution was mainly characterized by three bands. One main band in the visible region was shown at 485 nm originating from an extended chromophore, comprising both aromatic rings, connected through the azo bond. The other two bands in the ultraviolet region located at 229 nm and 310 nm were associated with benzene-like structures.
in the molecule (Yang et al., 2011; Wu et al., 2012). The disappearance of the visible band with the reaction time was correlated with the fragmentation of the azo links which led to the decolorization of AO7. As the reaction proceeded, the absorbance peak at 229 and 310 nm also diminished, indicating that the benzene and naphthalene rings were degraded to smaller aliphatic compounds and eventually CO₂ and water. To verify the mineralization of AO7, the values of TOC before and after treatment were monitored. As shown in Fig. 7b, the TOC removal efficiency achieved 56.5% after 60 min reaction.

3 Conclusions

This study showed that the combination of ultrasound with heterogeneous activation process is effective for the decolorization of AO7. The bimetallic Fe-Co/GAC catalyst behaves as efficient and stable catalyst for the heterogeneous oxidation of AO7 in the presence of ultrasonic irradiation. The optimal conditions for efficient AO7 degradation were pH 5.8, PS concentration 0.5 g/L, catalyst loading 0.8 g/L, stirring rate 500 r/min, and ultrasonic power 60 W. Under the conditions above, the decolorization efficiency was 98.3% and TOC removal was 56.5% within 60 min.

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- **Daqiang Yin**  
  Tongji University, China
- **Zhongtang Yu**  
  The Ohio State University, USA

### Municipal solid waste and green chemistry

- **Pinjing He**  
  Tongji University, China
- **Daqiang Yin**  
  The Ohio State University, USA

### Environmental ecology

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  Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, China

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