

CuFe₂O₄ as an Efficient Peroxymonosulfate (PMS) Activator for Sulfamethoxazole (SMX) Removal via Singlet Oxygen in Sewage

Zhe Li^a, Keke Zhi^{a,*}, Bohong Wang^b, Lianghui Guo^a

^aState Key Laboratory of Heavy Oil Processing-Karamay Branch, China University of Petroleum-Beijing at Karamay, Karamay 834000, PR China

^bNational & Local Joint Engineering Research Center of Harbor Oil & Gas Storage and Transportation Technology, Zhejiang Ocean University, No.1, Haida South Road, 316022, Zhoushan, P.R. China
 zhikeke@cupk.edu.cn

During the COVID-19 pandemic, antibiotic production and consumption are increasing dramatically, and exploring the effective treatment of medical wastewater methods has become a major challenge to environmental protection. SMX as an important sulfonamide antibiotic (SA) has been frequently observed worldwide in contaminated water. CuFe₂O₄, as a class of promising efficient catalysts to activated PMS has been widely used to degrade organic pollutants. To maximize the catalytic performance of none-support-CuFe₂O₄, a single-needle electrospinning approach in conjunction with the sol-gel process at 500 °C is employed to synthesize CuFe₂O₄-500 (CFO-500), which was used for PMS activation to degrade SMX. The results indicate that CFO-500 exhibited an excellent degradation rate on SMX in 90 min, which exceeded 94.63 %. The outstanding properties could be mainly ascribed to two aspects: (i) Successfully obtained almost singular copper ferrite phases, which have higher specific surface area and more electron transfer path; (ii) The cooperation of free radical and singlet oxygen, abundant active sites, and excellent electron transfer capability make it a leap in the ability to produce active substances. ·SO₄⁻ and singlet oxygen ¹O₂ played a dominant role according to EPR and quenching tests, especially ¹O₂. This work may provide a new idea and mechanism for activating PMS to degrade wastewater.

1. Introduction

Sustainable development has become the cornerstone of building a community with a shared future for mankind (Klemeš et al., 2022). Cleaner production is the most important part in sustainable development, aiming to reduce greenhouse gas (Zhi et al., 2023), effluents and contamination generation (Wang et al., 2022). Since the end of 2019, pneumonia caused by COVID-19 has been spread all over the world. The useable of antibiotics for viral infections are increasing dramatically (Nandi et al., 2023). Antibiotics have been widely detected in many regions of the world, especially in medical sewage (Zou et al., 2022), urban (Lyu et al., 2023), and agricultural wastewater (Franklin et al., 2022). The indiscriminate discharge of sewage will lead to the increase of antimicrobial resistance (AMR) due to incomplete metabolism, which estimated that 4.95 M deaths worldwide in 2019 were related to AMR, of which 1.27 M deaths were directly attributed to AMR (Murray et al., 2022). Therefore, antibiotic pollution has become an urgent issue of public health and environmental safety.

SMX as the most representative sulfonamide antibiotic, is widely used in aquaculture (Liang et al., 2021), human clinical therapy (Liang et al., 2018), and animal husbandry (Liu et al., 2022). Because of its extensive use, SMX has become the most frequently detected antibiotic in wastewater, whose concentration is up to 63 µg/L in farm runoffs in China (Wei et al., 2011). Owing to the low biodegradability and long half-life (81.5 days) (Ma et al., 2019), SMX can last for a long time under natural conditions, and thus was categorized as a persistent antibiotic. Low concentration of SMX could already promote antibiotic resistance through a selection of naturally resistant bacteria in just 30 days, and thus the potential risk to human health. It is crucial to find an efficient way to degrade SMX (Zhang et al., 2023).

Various treatment methods, including photolysis (Ahmed et al., 2014), adsorption (Zeng and Kan 2020), piezoelectric catalytic activation (Xu et al., 2021), and advanced oxidation processes (AOPs) (Zhi et al., 2021) have been applied for eliminating SMX from water. AOPs are high-profile technology to destroy organic pollutants by highly oxidizing agents. AOPs based on sulfate radical ($\bullet\text{SO}_4^-$) (SR-AOPs), owing to the high redox potential ($E^\circ(\bullet\text{SO}_4^- / \text{SO}_4^{2-}) = 2.5 - 3.1 \text{ V}$) (Neta et al., 1988) of the $\bullet\text{SO}_4^-$, be similar as hydroxyl radicals ($\bullet\text{OH}$) ($E^\circ(\bullet\text{OH} / \text{H}_2\text{O}) = 1.9 - 2.7 \text{ V}$) (Buxton et al., 1988) are gaining more interest. Transition metals were confirmed for the activation of PMS and the generation of inorganic radical species such as $\bullet\text{SO}_4^-$ and $\bullet\text{OH}$ (Anipsitakis and Dionysiou 2004). Copper ions have shown excellent performance in $\text{Cu}^{2+} / \text{PMS}$ system due to Cu could decompose PMS smoothly and Cu^{3+} could directly oxidize the organic pollutants (Feng et al., 2017). Besides, Fe(III)-to-Fe(II) cycling can transfer an electron to PMS, causing the breakage of O-O bond and the generation of radicals (He et al., 2022). However, the above homogeneous catalysis were difficult to retrieve, which may lead to secondary pollution of water (Karimian et al., 2020).

Singlet oxygen has been the most concerned active oxygen species in recent years (El-Naggar et al., 2023). It is widely proposed that it plays a decisive role in the process of transition-metal compounds activating PMS to degrade organic pollutants (Li et al., 2021). Due to its strong electrophilic ability (Zeinali et al., 2019), it is usually used to attack the rich electronic group in the pollutants (Peng et al., 2022), decompose the macromolecular organic pollutants, gradually mineralize, and finally generate water and carbon dioxide.

In recent years, magnetic ferrite spinel material with easy separation, recovery, high stability, low toxicity and low-mental ions leakage (Peng et al., 2022), has been widely used to active PMS in order to degrade organic pollutants (Gao et al., 2020). CuFe_2O_4 is an important class of magnetic materials with a ferrite spinel structure. Spinel oxides have long played an important role in various catalytic applications. Therefore, we used CuFe_2O_4 to catalyze PMS for degrading SMX. In this study, a cubic structure of CuFe_2O_4 has been successfully prepared, utilizing a single-needle electrospinning approach in conjunction with the sol-gel process at 500 °C employed to synthesize CuFe_2O_4 -500 (CFO-500), where all Fe^{3+} are located at the octahedral sites and Cu^{2+} at the tetrahedral sites. The paper also firstly demonstrates the effect of $^1\text{O}_2$ during the processing of SMX being decomposed into small molecules. By the structure, Fe(III)-to-Fe(II) and Cu(II)-to-Cu(III) cycling can provide ample electrons to PMS, thereby generating of radicals. CFO-500 exhibited an excellent degradation rate on SMX in 90 min, which exceeded 94.63 %.

2. Materials and methods

2.1 Chemical reagents

$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were purchased from Sinopharm Chemical Reagent Co., Ltd. Polyvinyl Pyrrolidone (PVP) was purchased from Shanghai Aladdin Bio-Chem Technology Co., LTD. Acetonitrile was HPLC grade and purchased from Thermo Fisher Scientific (China) Co., Ltd. Oxalic Acid was purchased from Shanghai Adamasi Reagents Co., Ltd. SMX, PMS, EtOH, Tert-Butanol, L-Histidine, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ and Na were all analytical grade and purchased from Shanghai Adamasi Reagents Co., Ltd. Deionized water was purified by a Millipore Milli-Q gradient system to HPLC grade in order to prepare all the above solutions.

2.2 Preparation of CFO-500

The magnetic polyporous CFO-T was made utilizing a single-needle electrospinning approach in conjunction with the sol-gel process (Jing et al., 2015). All of the reagents used in this study are chemically pure and have not been purified further. Two procedures were used to make the precursor for electrospinning. First, a transparent solution was made by dissolving about 0.242 g $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and about 0.808 g $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in a 10 mL binary co-solvent of N,N-dimethyl formamide (DMF) and ethanol at a 1:1 ratio for 2 hours at room temperature under strong magnetic stirring. Second, about 0.764 g polyvinylpyrrolidone (PVP, Mw = 1,300,000) was added to the 8.786 g nitrate solution extracted from the aforementioned solution, and a transparent nitrate/PVP sol was formed after stirring for about 8 hours. A certain volume of nitrate/PVP sol was held in a glass syringe equipped with a flat-tipped stain-less needle acting as a spinneret in a typical electrospinning procedure. The needle was coupled to a +14 kV voltage supplied by a DC high-voltage source, with an inner diameter of around 0.4 mm. A steel frame was put about 14.5 cm below the tip of the needle for collecting the CFO-500. A micro-injection pump kept the spinning rate at 0.4 mL/h. After the electrospinning was completed, a non-woven nitrate/PVP fibrous membrane was transported from the collector to a muffle and annealed for 3 h at 500 °C, whose heating rate was 60 °C h⁻¹.

2.3 Catalytic degradation experiment

Five sample systems (TBA, EtOH, L-Histidine, Nature, and None) were prepared for an intensive study on the degradation activity of CFO-500. The reduction of SMX by CFO-500 in the presence of PMS was carried out in a 100 mL conical bottle. Firstly, A total of 50 mL SMX_(aq) (pH=9, 10 mg /L), 0.8 mM TBA (quenching $\bullet\text{SO}_4^-$,

EtOH quenching $\cdot\text{SO}_4^-$ and $\cdot\text{OH}$, L-Histidine quenching $^1\text{O}_2$) and 10 mg CFO-500 were mixed, then put them in a shaker (170 RPM, 25 °C) shaking for 30 min so as to evenly disperse the catalyst in the medium. Secondly, inject 1 mL solution into the corresponding liquid phase vials and mark them as 0 min. Thirdly, 5 mg PMS was introduced to start the degradation of SMX. At a given reaction time interval, 1 mL of the aqueous solution was sampled and filtered with a filter (0.22 μm), then instantly quenched with excessive $\text{Na}_2\text{S}_2\text{O}_3$ solution. In the blank control group 1 (Nature Group), the first step only adds 50 mL SMX. Other steps are the same as above. In the blank control group 2 (None Group), expect that no quencher is added. other steps are the same as above. Each group of experiments was performed three times to obtain the average value and standard error.

2.4 Test method

The degradation activity of CFO-500 was investigated using SMX as a chemical probe, which used High-Performance Liquid Chromatography (HPLC) as a detection instrument. HPLC (PERSEE) with a VWD detector and symmetry C_{18} column (5 μm 250 \times 4.6 mm, pgeneral) are used to determine the concentration of SMX at a wavelength of 264 nm. The flow rate of the mobile phase was 1 mL min^{-1} , which consisted of acetonitrile and 10 mM oxalate_(aq) (40:60, v/v). Then, the standard curve was used to calculate the content, and the calculation formula of SMX degradation rate as the below:

$$\eta = \frac{(C_0 - C_t)}{C_0} \quad \ln \frac{C_0}{C_t} = kt \quad (1)$$

The kinetic data were fitted with a Pseudo-First-Order kinetic model to compare the SMX degradation kinetic rates, Where C_0 is the initial concentration (mg L^{-1}), C_t is the concentration at time t (mg L^{-1}), η is the degradation rate and k (min^{-1}) is the rate constant for the pseudo-first-order kinetic models.

3. Result and discussion

Determine the Standard Curve of SMX by HPLC, the standard curve equation are presented as follows:

$$Y = 0.0000070736X - 0.050493 \quad (2)$$

Where Y means the calculated content (mg/L), X means the Peak area μAuxs of VWD detector, respectively. The catalytic SMX degradation kinetics were conducted to study the degradation performance of prepared catalysts. By fitting the kinetic data with a first-order kinetic model, we can know which active substance exhibited the fastest catalytic performance among all active substances. After quenching $^1\text{O}_2$ in the system by L-Histidine, the degradation of SMX was dropped to about 50 % (Figure 1b). However, the degradation of SMX was not showing obvious changes by quenching $\cdot\text{SO}_4^-$ and $\cdot\text{OH}$ in Figure 1. Combined with Electron Paramagnetic Resonance (EPR) data, we can confirm that the major active substances are $^1\text{O}_2$. Through analysis of the kinetic constants of different active substances in Figure 1a, when $\cdot\text{SO}_4^-$ and $\cdot\text{OH}$ were quenched, the rate constant of reaction (k) of $^1\text{O}_2$ is about 0.025, while when $^1\text{O}_2$ were quenched, the k of $\cdot\text{SO}_4^-$ and $\cdot\text{OH}$ were only 0.01. This shows that the production rating of $^1\text{O}_2$ is faster, and its role in the reaction is earlier than $\cdot\text{SO}_4^-$ and $\cdot\text{OH}$. Then, combining with DFT calculations, ESP mapped molecular vdW surfaces, the degradation process of SMX is reasonably proposed.

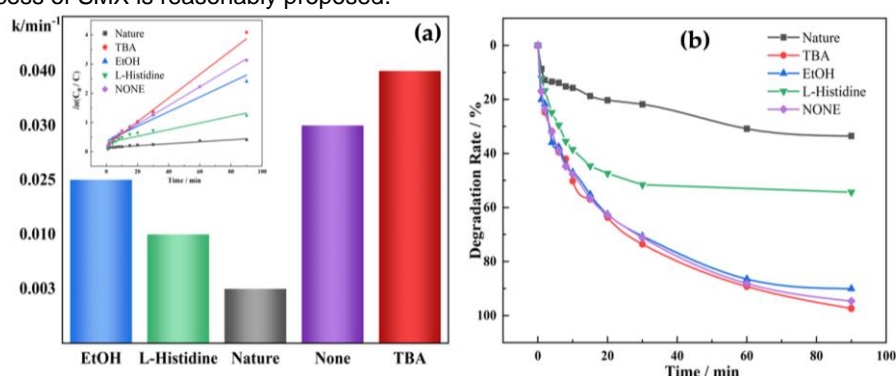


Figure 1: The catalytic performance of CFO-500: Influence of different active substances on CFO-500; (a) and kinetic constants (b)

Singlet oxygen ($^1\text{O}_2$) as an electrophilic reagent can attack electron-rich groups. Figure 3 shows the surface electrostatic potential of SMX, which was analysed by DFT calculations and their ESP-mapped molecular vdW

surfaces (Figure 3c). It is not difficult to see that electrophilic reagents can attack –SN– bond in SMX easily. The reaction of the degradation processing is shown in Figure 2.

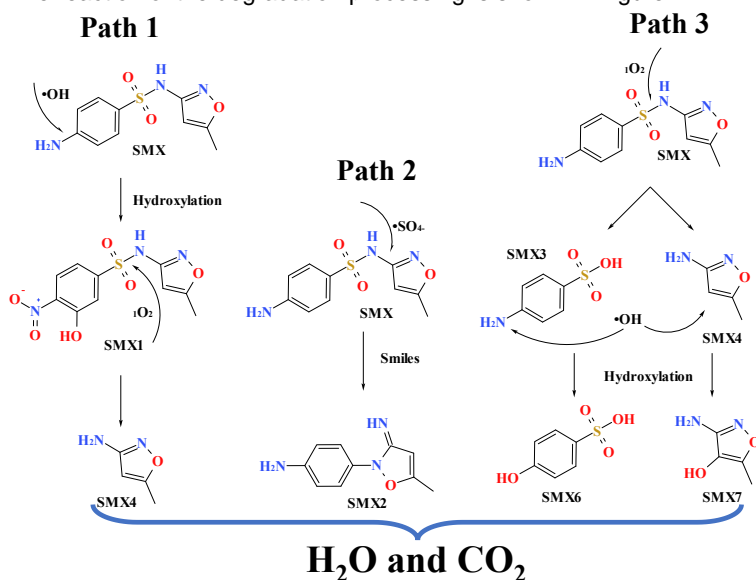


Figure 2: The inferred degradation route of SMX

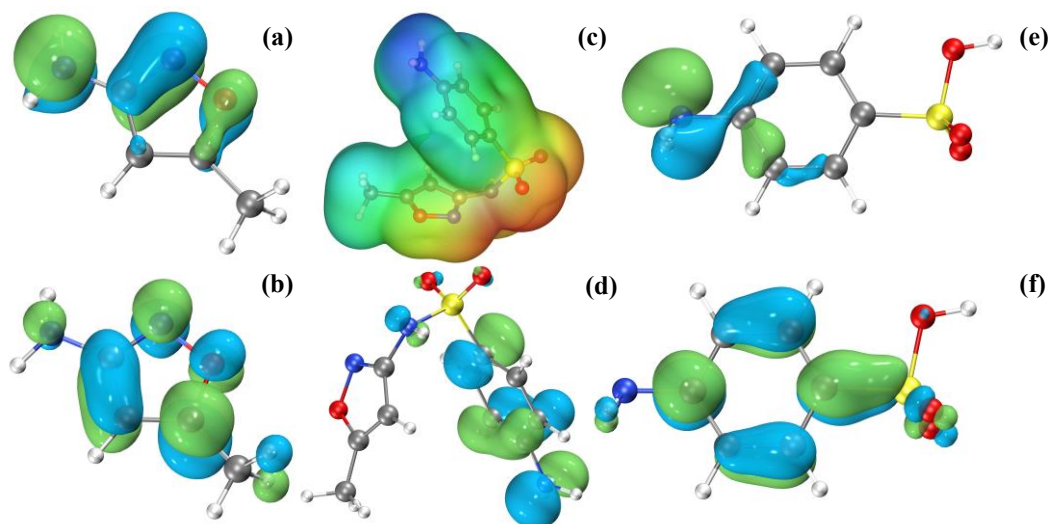


Figure 3: HOMO (a), LUMO orbital isosurface of SMX4 (b). ESP mapped molecular vdW surface of SMX (c), HOMO orbital isosurface of SMX (d). HOMO (e), LUMO orbital isosurface of SMX3 (f)

Analysis of Path 1, $\cdot\text{OH}$ attack - NH_2 on the benzene ring, because the N atom of - NH_2 in SMX had the greatest positive electricity. Therefore, the electrophilic reaction occurred to form 4-hydroxy-SMX. Then the 4-hydroxy-SMX was oxidized by $\cdot\text{OH}$ to form SMX1 (Liang et al., 2022). Then, $^1\text{O}_2$ will attack the –SN– bond of SMX1 to form SMX4. Analysis of Path 2, the SMX2 was an SO_2 extrusion product (SEP). The formation of SEP is usually accompanied by a Smiles rearrangement reaction (Ji et al., 2017). As shown in Figure 3a, b, e and f, the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) of SMX3 and SMX4 were used to predict their active sites of them. It can be found that the LUMO and HOMO were mainly concentrated on the aromatic ring of SMX4 and the five-membered ring of SMX3, indicating that it tended to react with $\cdot\text{SO}_4^-$ and $\cdot\text{OH}$. However, there is no HOMO concentrated on the five-membered rings in SMX (Figure 3d). Because of this, in Path 3, when the $^1\text{O}_2$ attacks the –SN– bond of SMX (Yi et al., 2023), the active site of the product will increase, which can maximize the $\cdot\text{SO}_4^-$ and $\cdot\text{OH}$ role. Due to the high-water solubility of SMX3 and its high reactivity with $\cdot\text{SO}_4^-$, SMX3 will soon be decomposed into small molecules and finally, become water and carbon dioxide.

4. Conclusions

Main conclusions can be drawn from this work: Combined with LUMO, HOMO and ESP mapped molecular vdW surface, the influence of three different active substances during the degradation process were analysed. Firstly, the molecular structure was split by $^1\text{O}_2$, which owing to its high electrophilic ability, can attack strong electron-donating groups, and gradually decompose macromolecular organics. Making the molecule become smaller molecular structures which were easy to be reacted by $\bullet\text{SO}_4^-$ and $\bullet\text{OH}$. Under the joint action of $^1\text{O}_2$, $\bullet\text{SO}_4^-$ and $\bullet\text{OH}$, the molecule gradually becomes H_2O and CO_2 . On this basis, we conclude that $^1\text{O}_2$ has played an important role in this system. Its contribution is about 50 %, and the removal of SMX can reach 94.63 % in 90 min. Future research could examine the different structures of CuFe_2O_4 , which are synthesized at different temperatures. In addition, more about $^1\text{O}_2$ mechanization might prove an important area for future research.

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