Chinese Chemical Letters 31 (2020) 2831–2834

Contents lists available at ScienceDirect

Chinese Chemical Letters

journal homepage: www.elsevier.com/locate/cclet

Communication

New insight in the O_2 activation by nano Fe/Cu bimetals: The synergistic role of Cu(0) and Fe(II)

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ARTICLE INFO

Article history:
Received 15 June 2020
Received in revised form 10 July 2020
Accepted 5 August 2020
Available online 7 August 2020

Keywords:
Advanced oxidation
Nano Fe/Cu bimetals
Oxygen activation
Hydroxyl radical
Heterogeneous catalysis

ABSTRACT

This study demonstrated that as-synthesized nano Fe/Cu bimetals could achieve significant enhancement in the degradation of diclofenac (DCF), as compared to much slow removal of DCF by Cu(II) or zero valent iron nanoparticles (nZVI), respectively. Further observations on the evolution of O_2 activation process by nano Fe/Cu bimetals was conducted stretching to the preparation phase (started by nZVI/Cu^{2+}). Interesting breakpoints were observed with obvious sudden increase in the DCF degradation efficiency and decrease in solution pH, as the original nZVI just consumed up to Fe(II) and Cu(II) appeared again. It suggested that the four-electrons reaction of O_2 and Cu-deposited nZVI would occur to generate water prior to the breakpoints, while Cu(0) and Fe(II) would play most important role in activation of O_2 afterwards. Through the electron spin resonance (ESR) analysis and quenching experiments, $^{\bullet}$OH was identified as the responsible reactive species. Further time-dependent quantifications in the cases of Cu(0)/Fe(II) systems were carried out. It was found that the $^{\bullet}$OH accumulation was positively and linearly correlated with nCu dose, Fe(II) consumption, and Fe(II) dose, respectively. Since either Cu(0) or Fe(II) would be inefficient in activating oxygen to produce $^{\bullet}$OH, a stage-evolution mechanism of O_2 activated by nano Fe/Cu bimetals was proposed involving: (a) Rapid consumption of Fe(0) and release of Fe(II) based on the Cu-Fe galvanic corrosion, (b) adsorption and transformation of O_2 to O_2^{2-} at the nCu surface, and (c) Fe(II)-catalyzed activation of the adsorbed O_2^{2-} to $^{\bullet}$OH.

Diclofenac (DCF), one of the anti-inflammatory agents, is widely used in the modern society [1]. As a consequence, large amount of this compound and its derivatives are liberated into municipal water treatment systems. However, it is known that biological process or adsorption is not effective enough to remove most of pharmaceuticals and personal care products (PPCPs) [2]. Numerous studies reported the existence of DCF in the ecosystems and its negative effect on aquatic flora and fauna [3]. In these decades, advanced oxidation processes (AOPs) are acknowledged as the most applicable technologies to remove PPCPs [4]. It takes advantage of rapidity, stability and nonselectivity, but simultaneously suffers from high cost and secondary contamination [5,6]. Among of the AOPs, zero-valent-iron (ZVI) based catalyzed oxidation is famous because of iron's cheapness and non-toxicity [7]. Nevertheless, the addition of oxidants such as H_2O_2, peroxydisulfate (PDS) and peroxymonosulfate (PMS) still make these technologies less economically desirable. Therefore, green and inexpensive oxidant would be acclaimed to boost the ZVI based technologies.

Recently, it was reported reactive oxygen species (ROS) could be produced when ZVI corroded in oxic environment [8]. This system was attracting that oxygen could be utilized to produce oxidant instead of external addition. The generation of ROS originated from the electrons transfer between iron (Fe(0) and Fe(II)) and oxygen *via* two possible pathways. The direct transfer of two electrons from Fe(0) to O_2 would lead to the production of H_2O_2 (Eq. 1), which could further react with Fe(II) to generate hydroxyl radicals ($^{\bullet}$OH) (Eq. 2) [9]. Besides, oxygen could also obtain one electron from Fe(II) in twice successively to produce superoxide radicals ($O_2^{\bullet-}$) and H_2O_2, respectively (Eqs. 3 and 4) [8]. However, although nano ZVI (nZVI) was able to activate oxygen to produce $^{\bullet}$OH and degrade organics, both the electron efficiency and observed reaction rate were low. It might be attributed to two possible

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https://doi.org/10.1016/j.cclet.2020.08.006

reasons, *i.e.* the side reaction of quadruple-electron transfer between Fe(0) and oxygen (Eq. 5) [10], and the iron surface passivation during corrosion [11].

$$\mathrm{Fe(0)} + \mathrm{O_2} + 2\mathrm{H^+} \rightarrow \mathrm{Fe^{2+}} + \mathrm{H_2O_2} \tag{1}$$

$$\mathrm{Fe^{2+}} + \mathrm{H_2O_2} \rightarrow \mathrm{Fe^{3+}} + {}^{\bullet}\mathrm{OH} + \mathrm{OH^-} \tag{2}$$

$$\mathrm{O_2} + \mathrm{Fe^{2+}} \rightarrow \mathrm{O_2}^{\bullet -} + \mathrm{Fe^{3+}} \tag{3}$$

$$\mathrm{O_2}^{\bullet -} + \mathrm{Fe^{2+}} + 2\mathrm{H^+} \rightarrow \mathrm{H_2O_2} + \mathrm{Fe^{3+}} \tag{4}$$

$$\mathrm{O_2} + 2\mathrm{Fe(0)} + 4\mathrm{H^+} \rightarrow 2\mathrm{H_2O} + 2\mathrm{Fe^{2+}} \tag{5}$$

The deposition of other transition metal on ZVI was proposed as an effective method to enhance its reactivity. As one of metal ions frequently detected in water/wastewater, copper was usually used as a catalyst to improve the reactivity of ZVI. It was reported that the pollutants reduction by Cu/Fe bimetals was much more rapid than iron alone [12]. The reason was proposed at two aspects that copper would facilitate the generation/adsroption of activated atomic hydrogen and galvanic cell formed between Fe-Cu would enhance the corrosion of iron. In recent years, several studies have also investigated the performance of Cu/Fe bimetals on oxygen activation [13–17]. However, contradictory results and controversial conclusions were given by the researchers. Jia *et al.* found that the addition of copper ions would significantly promote the degradation of atrazine by nano iron wires [13]. They proposed that added $\mathrm{Cu^{2+}}$ would rapidly deposit on iron surface to form Cu/Fe bimetal, followed by the accelerated corrosion of iron. The enhanced liberation of $\mathrm{Fe^{2+}}$ during corrosion facilitated the single-electron activation of oxygen and production of $^{\bullet}\mathrm{OH}$. Luo *et al.* found the Cu-doped Fe@$\mathrm{Fe_2O_3}$ was a good catalyst in electro-Fenton process [14]. They thought the doped Cu(0) not only accelerated the iron cycle, but also effectively activate oxygen with copper cycle *per se*. Zhao *et al.* found iron-copper embedded carbon aerogel (FeCuC) has high reactivity for oxygen reduction reaction when it was used as the cathode in electro-Fenton system, and doped Cu enhanced the selectivity of double-electron reduction of $\mathrm{O_2}$ [15,16]. Nevertheless, contrary results were found by Yamaguchi *et al.* who investigated the relationship between Cu/Fe (w/w) ratio and $^{\bullet}\mathrm{OH}$ [17]. Their results showed the existence of Cu blocked the generation of $^{\bullet}\mathrm{OH}$. Obviously, Cu/Fe bimetal exhibited disparate performance compared with iron in the case of oxygen activation, while its synergistic effect waited for systematic investigation and the mechanism needed further clarification yet.

In this study, nano Fe/Cu bimetals was fabricated *in situ via* instant $\mathrm{Cu^{2+}}$ reduction by nZVI. Different doses of Cu and Fe were investigated. DCF degradation, $^{\bullet}\mathrm{OH}$ production and metal ions evolution were simultaneously quantified during the reaction. The main objectives were to evaluate the ability of nano Fe/Cu bimetals to activating $\mathrm{O_2}$, and reveal the mechanism based on the synergistic role of Fe and Cu species.

nZVI and nano Cu particles (nCu) were synthesized *via* the sodium borohydride reduction [18]. All nZVI and nCu were used as prepared. nZVI exhibited a Fe(0)@$\mathrm{Fe_xO_y}$ core-shell structure [19] and the thickness of the shell was about 3–4 nm (Fig. S1 in Supporting information). The deposition of Cu on nZVI (to generate nano Fe/Cu bimetals) was conducted at the inception of oxygen activation experiments. The *in situ* fabrication of bimetal was because of two reasons. The reduction of $\mathrm{Cu^{2+}}$ on nZVI was so rapid that concentration of Cu ions would decrease to below the detection limit in seconds. In addition, the nano Cu/Fe bimetals of ultra-high reactivity would intensely reacted with air, *i.e.* combustion. The characterization fabricated nano Cu/Fe bimetals could be seen in Fig. S2 (Supporting information). X-ray photoelectron spectroscopy (XPS) confirmed the deposition of Cu on nZVI, and the transmission electron microscopy (TEM) picture showed that the core-shell structure was not obviously broken by Cu deposition. The actual ratio of Fe/Cu for the catalysts could be seen in Table S1 (Supporting information).

All experiments were conducted at least duplicates in a series of 20 mL serum bottles placed in a rotary shaker with 200 rpm. In a typical degradation experiment, the serum bottles were open to air and filled with 10 mL solution containing predetermined amounts of DCF and $\mathrm{Cu^{2+}}$ ions. The reaction was initiated by the addition of nZVI. At regular intervals, sacrificed bottles were taken out and the reaction was stopped by methanol or KI (TOC measurement). Then the water samples were immediately filtered by 0.22 μm PES membrane (Titan) and sent for analysis. In the experiment of nCu/Fe(II) system, the working solution contained 50 mmol/L dimethylsulfoxide (DMSO) and predetermined concentration of Fe(II) ions, and reaction was initiated by the addition of nCu.

DCF was quantified by a high-performance liquid chromatography (HPLC, LC-15C, Shimadzu) equipped with a UV–vis detector and a C18 column (Agilent, 5 μm, 4.6 × 150 mm). The mobile phase was a mixture of 0.1% (v/v) acetic acid (40%) and acetonitrile (60%), with a flow rate of 0.7 mL/min. The detection wavelength was set at 270 nm while the temperature was controlled at 35 °C. Total organic carbon was measured by a total organic carbon (TOC) analyzer (TOC-L CPH, Shimadzu). Aqueous Fe(II) and Cu(II) were spectrometrically measured employing an UV–vis spectrophotometer (UV-2600, Shimadzu), according to the neocuproine methods [20] and *o*-phenanthroline colorimetric method [21], respectively. The quantification of $^{\bullet}\mathrm{OH}$ based on the method described by Sahni *et al.* [22] and modified to mask the interference of Fe(II). Briefly, 1 mL bipyridyl (BPY) solution (20 mmol/L, with the solvent of acetonitrile:water = 1:1), 2 mL phosphate buffer (pH 4, 0.5 mol/L) and 0.5 mL dinitrophenylhydrazine (DNPH) solution (6 mmol/L, with the solvent of acetonitrile) were successively mixed with 0.2 mL filtered sample. Then, the mixture was kept under 40 °C for 90 min, ensuring the formation of HCHO-DNPH complex. Finally, HPLC was used for quantification of HCHO-DNPH at 355 nm, and the mobile phase was the same as that in the quantification of DCF.

Fig. 1a shows the comparative degradation of DCF in nZVI, Cu(II) and nZVI/Cu(II) systems under oxic environment. Cu(II) alone could not remove DCF, while only 7% DCF was removed by nZVI in 2 h. It indicated the oxygen activation by nZVI occurred with low efficiency, similar to previous researches [13]. Nevertheless, effective degradation of DCF realized in the nZVI/Cu(II) system, in which the removal ratio of DCF achieved 96% in 2 h. It was noted that Cu(II) disappeared in seconds after the mixture with nZVI, indicating its instant reduction on iron surface. Therefore, the

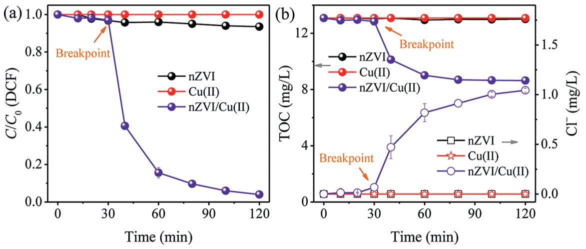

Fig. 1. The comparative (a) degradation of DCF, (b) TOC removal and $\mathrm{Cl^-}$ evolution in the nZVI, Cu(II) and nZVI/Cu(II) system. Initial conditions were: 10 mmol/L nZVI, 1 mmol/L Cu(II), 20 mg/L DCF, pH 6 and 25 °C.

solid catalyst in the nZVI/Cu(II) system could be considered as nano Fe/Cu bimetals, which obviously exhibited high reactivity for DCF removal. The experiment with N_2 sparging in the nZVI/Cu(II) system leaded to little degradation of DCF (Fig. S3 in Supporting information), excluding the direct reduction of DCF. Further measurement of TOC and chloride ions (Fig. 1b) evidenced the oxidation of DCF, indicating the oxygen activation occurred with high efficiency catalyzed by nano Fe/Cu bimetals. More interestingly, a breakpoint at about 30 min was observed in the nZVI/Cu(II) system. It was not until the breakpoint that the oxidation rapidly proceeded. Simultaneous analysis of aqueous Cu(II) showed that the liberation of Cu(II) occurred just after the breakpoint (Fig. S4a in Supporting information). It indicated the breakpoint probably agreed with total consumption of Fe(0), since oxidation of Cu(0) hardly happened at the presence of Fe(0). The concentration of aqueous Fe(II) underwent a "M-like" variation (Fig. S4b in Supporting information). The first rapid increase of aqueous Fe(II) concentration was attributed to the instant oxidation of Fe(0) by Cu(II). After that, aqueous Fe(II) might be apt to hydrolyze with pH climbing during iron corrosion. It was not until the breakpoint Fe(II) hydroxide redissolved with the sharp decrease of pH due to low-weight-acid production from DCF degradation. The following slow decrease of aqueous Fe(II) might be caused by oxidation by oxygen.

To further explore the key factors affecting the degradation of DCF and appearance of the breakpoint, Cu(II) and nZVI doses were investigated. Fig. S5 (Supporting information) shows the comparative removal of DCF as well as evolution of pH, aqueous Cu(II) and Fe(II), with different of Cu(II) doses. The increase of Cu(II) dose obviously elevated the removal ratio of DCF, while the breakpoint was negligibly affected (Fig. 2a). It supported the postulation that the breakpoint should be primarily dependent on Fe(0) consumption instead of Cu amounts, and the latter significantly affected efficiency of oxygen activation after the breakpoint. The pH of solution increased firstly with the rapid corrosion of iron, while began to decrease sharply around the breakpoint with significant degradation of DCF. The terminal pH was lower with more addition of Cu(II), that was accorded with the case of DCF degradation. Aqueous Cu(II) appeared around the breakpoint, regardless of the dose of Cu(II). However, the concentration of aqueous Cu(II) after the breakpoint was higher with more addition of Cu(II). It indicated Cu(0) would be consumed with DCF degradation. The concentration of aqueous Fe(II) was positively affected by the increase of Cu(II) dose. It should be resulted from more amounts of galvanic couples and lower pH. From Fig. S6 (Supporting information), the influence of nZVI dose on DCF degradation could be seen. The increase of nZVI dose slightly raised the removal of DCF, but significantly prolong the appearance of breakpoint (Fig. 2b). It further evidenced that breakpoint was because of the presence of Fe(0), since the more nZVI addition would lead to longer duration for total consumption of Fe(0). In addition, the negligible effect of nZVI dose on DCF degradation indicated Fe(0) did not directly contributed to the oxygen activation. The simultaneous evolution of pH, aqueous Cu(II) and Fe(II) was accorded with the degradation of DCF, and breakpoints could be obviously observed.

At this point, it could be concluded that Cu(II) deposition on nZVI, or nano Fe/Cu bimetals, could effectively oxidize DCF, and a breakpoint existed depending on the duration for total consumption of Fe(0). However, it was still unknown about the mechanism for enhanced degradation of DCF after the breakpoint. Several previous studies found Cu/Fe bimetal (or Cu(II) addition to nZVI system) would produce much more $^{\bullet}$OH *via* oxygen activation than ZVI alone system, while there were also reports thought that Cu deposition would inhibit $^{\bullet}$OH production. In this study the responsible oxidant was also qualitatively and quantitatively examined. The electron spin resonance (ESR) spectra shown in Fig. S7a (Supporting information) clearly indicated the $^{\bullet}$OH generation in nZVI/Cu(II) system through the appearance of four peaks with the intensity of 1:2:2:1, which could not be distinguished clearly in the nZVI or Cu(II) system. Since DMSO could capture $^{\bullet}$OH, the effect of DMSO addition was examined as shown in Fig. S7b (Supporting information). Significant inhibition was found in nZVI and nZVI/Cu(II) systems. It indicated the promoted degradation of DCF should be attributed to enhanced generation of $^{\bullet}$OH. This was confirmed by Figs. S7c-d (Supporting information), in which the production of $^{\bullet}$OH was positively related to Cu(II) addition.

Therefore, the effective degradation of DCF in the nZVI/Cu(II) (nano Fe/Cu bimetals) system resulted from the enhanced generation of $^{\bullet}$OH when Fe(0) was totally consumed. In this stage, the inorganic catalysts was constituted by Cu(0), Fe oxides and aqueous Fe(II) species. The increase of Cu(II) dose elevated both the amounts of Cu(0) and concentration of aqueous Fe(II). Obviously, the latter two catalyst were probably responsible for enhanced $^{\bullet}$OH production. It has been reported that Cu(0) could activate oxygen to produce $^{\bullet}$OH [14]. However, similar to our previous study, accumulation of $^{\bullet}$OH from oxygen activation by nCu alone was slow (Fig. S8a in Supporting information). Obviously, the rapid generation of $^{\bullet}$OH was not the result of oxygen activation by the presence of Cu(0) alone. There were also studies thought that the accelerated liberation of Fe(II) of Fe/Cu bimetal should be the primary reason for rapid generation of $^{\bullet}$OH [13]. Abundant amounts of Fe(II) ensured the rapid single-electron activation of oxygen. However, the experiment using Fe(II) alone showed that only about 30 μmol/L $^{\bullet}$OH would be produced in 2 h (Fig. S9a in Supporting information). Therefore, the enhanced generation of $^{\bullet}$OH should not be attributed to neither Cu(0) nor Fe(II) alone. Nevertheless, it was amazing that the coexistence of nCu and Fe(II) leaded to rapid production of $^{\bullet}$OH (Figs. S8 and S9 in Supporting information). Obviously, synergistic action with nCu and Fe(II) existed, which should be the reason for enhanced oxygen activation by nano Fe/Cu bimetals (nZVI/Cu(II) system) after the breakpoint. The dependence of $^{\bullet}$OH production on nCu and Fe(II) doses were further investigated, respectively. It could be seen

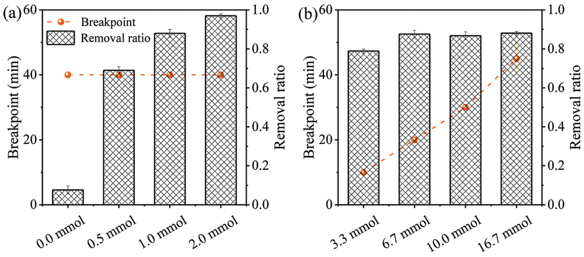

Fig. 2. The dependence of breakpoint and removal ration on (a) Cu(II) dose, and (b) nZVI dose. Initial conditions were: 13.4 mmol/L nZVI, 1 mmol/L Cu(II), 20 mg/L DCF, pH 6 and 25 °C.

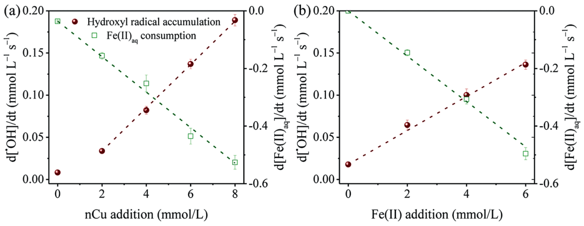

Fig. 3. The correlations of $^{\bullet}$OH production rate as well as Fe(II) consumption rate to (a) nCu dose, and (b) Fe(II) dose. Initial conditions were: 6 mmol/L Fe(II), 2 mmol/L nCu, 50 mmol/L DMSO, pH 6 and 25 °C.

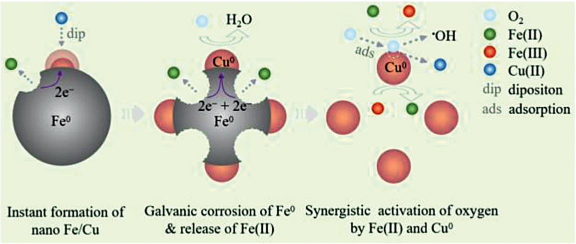

Fig. 4. Stage-evolution mechanism of oxygen activation by as-synthesized nano Fe/Cu bimetals.

clearly that $^{\bullet}OH$ production positively related to the doses of nCu and Fe(II) (Fig. 3).

It was previously reported that Cu(0) could promoted the cycle Fe(II)/Fe(III) [14]. In this study, the rapid reduction of Fe(III) by nCu was also found under the O_2-free circumstance, as shown in Fig. S10 (Supporting information). However, this was not enough to explain the role of Cu(0) because Fe(II) consumption positively related to nCu dose (Fig. 3a). Besides, it has been of considerable researches found oxygen could easily adsorb on Cu(111) and formed a peroxo form (O_2^{2-}) [23]. The later would likely to combine with protons to produce H_2O_2, or directly react with Fe(II) to produce $^{\bullet}OH$. It was evidenced by the result that the adsorbed O_2^{2-} instead of free H_2O_2 was detected in the system (Fig. S11 in Supporting information) and Fe(II) consumption rate linearly related to nCu dose (Fig S19 in Supporting information). Moreover, a strong correlation between Fe(II) consumption and $^{\bullet}OH$ production was also found (Fig. S12 in Supporting information). Therefore, it was probably that the oxygen adsorbed on the reactive sites of Cu surface would be apt to activated by Fe(II), producing the hydroxyl radical.

Based on the above discussion, a stage-evolution mechanism of O_2 activation by as-synthesized nano Fe/Cu bimetals was proposed as presented in Fig. 4. The instant reduction of Cu(II) onto Fe(0) surface to form the nano Fe/Cu bimetals would occur initially. Then the galvanic corrosion of Fe(0) proceeded with the quadruple-electron reduction of O_2 to H_2O. After the Fe(0) was consumed up, O_2 could be adsorbed on the surface of Cu nanoparticles and would be transformed into the peroxo form (O_2^{2-}) [23]. Simultaneously, the released Fe(II) could react with the adsorbed O_2^{2-} species, leading to the generation of $^{\bullet}OH$. The existence of Cu(0) could also lead to the reduction of Fe(III) to regenerate Fe(II), maintaining the Fe(II)/Fe(III) cycle.

To evaluate the applicability of the nano Fe/Cu bimetals, its reusability performance was also examined. As shown in Fig. S13 (Supporting information), the nano Fe/Cu bimetals could lead to 80% removal of DCF in 30 min even after three cycles. In addition, the reactivity of the catalyst could be almost totally recovered by nZVI reduction. Therefore, the nano Fe/Cu bimetals was a kind of catalyst of high reactivity for activating O_2 and was easy to be recycled.

Declaration of competing interest

The authors report no declarations of interest.

Acknowledgments

This study is financed support by the National Natural Science Foundation of China (Nos. 21677055 and 21407052), National Key Research and Development Program of China (No. 2019YFC1805204), Project of Three Gorges Corporation (No. JD-ZC-FW-20-001), and the Fundamental Research Funds for the Central Universities, HUST (No. 2017KFXKJC004). Huazhong University of Science & Technology Analytic and Testing Centre is thanked for the advanced analytic operations.

Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.cclet.2020.08.006.

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