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Effect of pipe corrosion product–goethite–on the formation of disinfection by-products during chlorination

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ABSTRACT

The effect of goethite on the formation of disinfection by-products (DBPs) during chlorination was investigated under various reaction times, pH, and chlorine concentrations. The experimental results indicated that in the presence of goethite, the formation of DBPs was enhanced as pH and chlorine concentration increased. The enhancement of DBP formation could be attributed to the generation of hydroxyl radical (·OH), which could oxidize natural organic matter (NOM) in water and produce more DBP precursors to react with chlorine. X-ray diffraction (XRD) and scanning electron microscope (SEM) analyses of goethite revealed that goethite may adsorb NOM on its surface and modify NOM structure. The reaction mechanisms of goethite-enhanced DBP formation were proposed in three steps: (1) the oxidation of NOM into small molecules available as DBP precursors by ·OH from the reaction between ferrous ions on goethite surface and chlorine; (2) the adsorption and modification of NOM on goethite surface responsible for increasing chlorine consumption; and (3) the presence of ferric ions on goethite surface promoted the formation of DBPs.

Keywords: Chlorination; Disinfection by-products (DBPs); Pipe corrosion; Nature organic matter (NOM); Hydroxyl radical; Ferric catalysis

1. Introduction

Chlorine has been used to disinfect drinking water for approximately 100 years because it is cheap and effective against micro-organisms, but it is accompanied with more than 600 disinfection by-products (DBPs) having been detected in chlorinated waters [1]. Many DBPs such as trihalomethanes (THMs), haloacetonitriles (HANs), halonitromethanes (HNMs), and N-nitrosodimethylamine (NDMA) have been reported to be carcinogenic to animals and are considered as probable human carcinogens [2–4]. Many studies have focused on the formation of DBPs in water treatment plants; however, little attention has been paid to the

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formation of DBPs in drinking water distribution systems.

In drinking water distribution systems, iron pipes could be oxidized by oxidizing agents such as oxygen and chlorine, leading to the oxidation of iron surface and production of corrosion products, including goethite, magnetite, lepidocrocite, sideritete, and green rust [5–7]. The release of metal ions in water could cause many problems, including the decrease in disinfection efficiency [8], the increase in DBP formation [7], and the deterioration in water quality [9].

Goethite, an iron oxyhydroxide, is the major pipe corrosion product in water distribution systems formed in aqueous environment at ambient temperatures [10,11]. Goethite shows strong uptake capacity for toxic metals [12] and oxyanions [13], which has been identified as an important sorbent for toxic ions in the environment [14]. Numerous studies have shown that the surface properties of goethite, especially the reactivity of OH groups on its surface, are important in determining the mobility, reversibility, and fate of adsorbed species [15,16]. Previous studies have indicated that goethite can convert H_2O_2 into $^{\cdot}OH$ through a surface-initiated chain reaction analogous to the Haber–Welss mechanism [17–20]. Because sodium hypochlorite (NaOCl) is also a strong oxidant like H_2O_2, goethite can convert NaOCl into $^{\cdot}OCl$ or produce $^{\cdot}OH$ [21]. Moreover, goethite can adsorb natural organic matter (NOM) onto its surface. The reaction between NaOCl and NOM may become easier on goethite surface so as to increase the formation of DBPs [22–24].

Since little attention has been paid on the role of goethite on DBP formation in drinking water distribution systems, the objectives of this research are (1) to systematically evaluate the presence of goethite on the formation of carbonaceous DBPs (C-DBPs) and nitrogenous DBPs (N-DBPs) at different solution pH values and reaction times and (2) to propose rational reaction mechanisms for goethite-enhanced DBP formation.

2. Materials and methods

2.1. Chemicals

All chemicals were at least of analytical grade except as noted. DBP standards for THMs, including chloroform (CF), bromodichloromethane (BDCM), dibromochloromethane (DBCM), and bromoform (BF) and HANs, including bromochloroacetonitrile (BCAN), dibromoacetonitrile (DBAN), dichloroacetonitrile (DCAN), trichloroacetonitrile (TCAN), and trichloronitromethane (TCNM) were purchased from Sigma–Aldrich (USA). Stock solutions of chlorine were prepared by NaOCl solution (available chlorine of 4–4.99%, Sigma–Aldrich, USA) and calibrated using the N,N-dethyl-p-phenylenediamine (DPD) colorimetric method [25].

Goethite (FeOOH, ~35% Fe) was also purchased from Sigma–Aldrich (USA). Sulfuric acid (H_2SO_4), sodium hydroxide (NaOH), anhydrous sodium sulfate (Na_2SO_4), and ammonia chloride (NH_4Cl) were purchased from Sinopharm Chemical Reagent Co., Ltd. (China) without further purification. Methyl *tert*-butyl ether (MtBE) and methanol were obtained from J.T. Baker (USA). All the solutions were prepared with ultra-pure water produced from a Milli-Q water purification system (Millipore, USA).

2.2. Water samples

Samples of the filtered water from filtration process at Yangshupu drinking water treatment plant (YDWTP) were collected in polypropylene containers, transported to the laboratory on ice, filtered through 0.45 μm cellulose membrane filters (Anpel Co., Ltd., Shanghai, China) to remove suspended solids upon arrival, and then stored at 4˚C in the dark. The characteristics of the filtered water are summarized in Table 1.

2.3. Experimental procedures

The experiments of DBP formation during chlorination were conducted in duplicate under headspace-free conditions in 40 mL glass screw-cap vials with PTFE-lined septa at controlled temperature (25 ± 1˚C) using the filtered water from YDWTP with or without the presence of goethite. Experimental parameters included solution pH (7–9) and reaction times (1–6 h and 24 h) with goethite concentration of 4 g/L. The chlorine concentration was fixed to 6 mg/L by adding suitable amount of stock NaOCl solution. All the

Table 1
Characteristics of the filtered water used in this study

Parameters	Values
pH	7.62
Total alkalinity (mg/L as $CaCO_3$)	92.5
Dissolved organic carbon (mg C/L)	1.69
Total dissolved organic nitrogen (mg N/L)	1.51
UV_{254} (cm^{-1})	0.043
Bromide (mg/L)	0.050
Chloride (mg/L)	63.3
Sulfate (mg/L)	45.6
Nitrate (mg N/L)	1.33
Iron (mg/L)	0.02
Manganese (mg/L)	0.05

samples were buffered using 10 mM phosphate solution and pH values were adjusted with either 0.01, 0.1, and 1 M H_2SO_4 or NaOH. After reaching the planned reaction time, samples were quenched using NH_4Cl instead of $Na_2S_2O_3$ to avoid the interaction with formed N-DBPs and filtered through 0.22 μm PTFE filters (CNW Technologies GmbH, Düsseldorf, Germany) to remove goethite and then extracted for the analysis of C- and N-DBPs as soon as possible. Two sets of control experiments were performed. The first set was done with direct measurement of C- and N-DBPs in the filtered water from YDWTP, and no detectable DBPs were found. The second set was done in the Milli-Q water dosed with chlorine and goethite, and DBPs were not detectable either.

2.4. Analytical methods

The method developed for quantifying C- and N-DBPs was similar to that of the US EPA Method 551.1 with minor modifications [26]. Samples were performed with liquid–liquid extraction using MtBE, and the extracts were then analyzed using a gas chromatograph (GC) (GC-2010, Shimadzu, Japan) equipped with an electron capture detector (ECD) and a HP-5 capillary column (30 × 0.25 mm i.d., 0.25 μm film thickness, J&W, USA). The detailed methods can be found in our pervious study [27].

Chlorine residuals were analyzed at the end of reaction time by DPD colorimetric method [25]. Solution pH was measured using a pH meter (FE20-FiveEasy, Mettler Toledo, Switzerland), which was calibrated regularly with standard buffer solutions (Mettler Toledo, USA).

Total organic concentration (TOC) of samples was measured using a Shimadzu TOC-VCSH analyzer (Shimadzu, Japan) and the detection limit was 0.1 mg C/L. UV_{254} was measured using a 1-cm quartz cell at a wavelength of 254 nm with a UV–Vis spectrophotometer (SQ-4802, UNICO, Shanghai). Inorganic anions were analyzed using an ion chromatography (Dionex ICS-2000, USA) equipped with a conductivity detector, a Dionex AS11-HC analytical column (250 × 4.0 mm i.d.), and a Dionex AG11-HC guard column (50 × 4.0 mm i.d.).

2.5. Characterization of goethite

In order to evaluate the crystallization of goethite after chlorination experiments, goethite samples were collected and analyzed using X-ray diffraction (XRD) with a Philips X′pert Pro MDP diffractometer (The Netherlands) equipped with a copper anticathode (Cu Kα1 radiation, λ = 1.5418 Å). XRD was operated with scanning angles of 5–80° in 4,488 increments with a 60 s dwell at each step. The collected patterns were compared with the database to identify the presence of mineral species.

The surface morphology of goethite samples was observed using a scanning electron microscope (SEM) (Philips XL30, The Netherlands) with accelerating voltages of 9–20 keV. Powdered samples were mounted on 12 mm diameter aluminum stubs using carbon adhesive pads and then coated with a layer of gold using a BAL-TEC SCD 004 Sputter Coater (Bal-Tec, Vaduz, Liechtenstein) before SEM observation.

3. Results and discussion

3.1. Effect of reaction times on DBP formation

The DBPs detected for all samples included three regulated C-DBPs (CF, BDCM, and DBCM) and three emerging N-DBPs (DCAN, BCAN, and TCNM). The concentrations of C-, N-DBPs, and residual chlorine as a function of reaction time are shown in Fig. 1(a)–(c), respectively. The concentrations of all species of C- and N-DBPs increased with the increase in reaction time with or without the presence of goethite (Fig. 1(a) and (b)). Because common hydraulic residence time in water distribution system is 24 h or greater for utility practices in large cities, the DBP formation at 24 h was also examined. In the presence of goethite, the concentration of CF, BDCM, and DBCM increased from 24.2, 11.7, and 5.4 μg/L after 1 h chlorination to 70.8, 19.7, and 17.8 μg/L after 24 h chlorination (Fig. 2(a)). The concentration of BCAN, DCAN, and TCNM increased from 10.1, 2.5, and 1.9 μg/L after 1 h chlorination to 39.1, 8.8, and 4.2 μg/L after 24 h chlorination (Fig. 2(b)). The increase in DBP formation was more profound at the early stage of chlorination with 66–100% C-DBPs and 70–79% N-DBPs, formed in the first 6 h compared to those at 24 h. In the presence of goethite, the formation of C- and N-DBPs was higher compared to that without the presence of goethite (Fig. 1(a) and (b) as well as Fig. 2(a) and (b), which is also the case for chlorine consumption (Fig. 1(c)).

It was reported that there would be three reactions in goethite–chlorine suspension solution and on goethite surface (shown in Eqs. (1)–(3)) [21,28–31]. From the results shown in Fig. 1(a) and (b), it is obvious that goethite can enhance the formation of C- and N-DBPs. The reactions between ferrous ion and $HOCl/OCl^-$ can produce a hydroxyl radical ($^{\cdot}OH$) (Eq. (3)), which can oxidize NOM to more

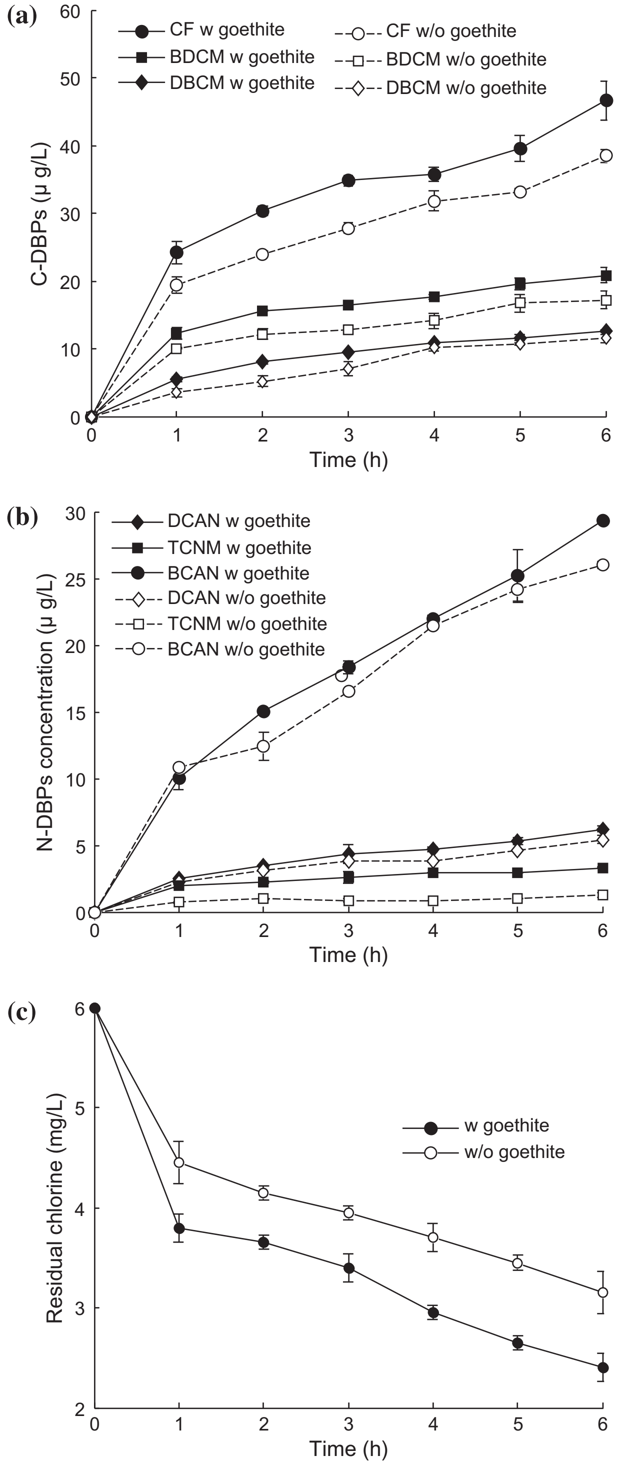

Fig. 1. The concentrations of C-DBPs (a), N-DBPs (b), and chlorine consumption (c) as a function of reaction time during chlorination with (w) and without (w/o) the presence of goethite. Temperature = 20 ± 1˚C, goethite concentration = 4 g/L, pH 7, Cl_2 concentration = 6 mg/L. Error bars represent one standard deviation of duplicate measurements.

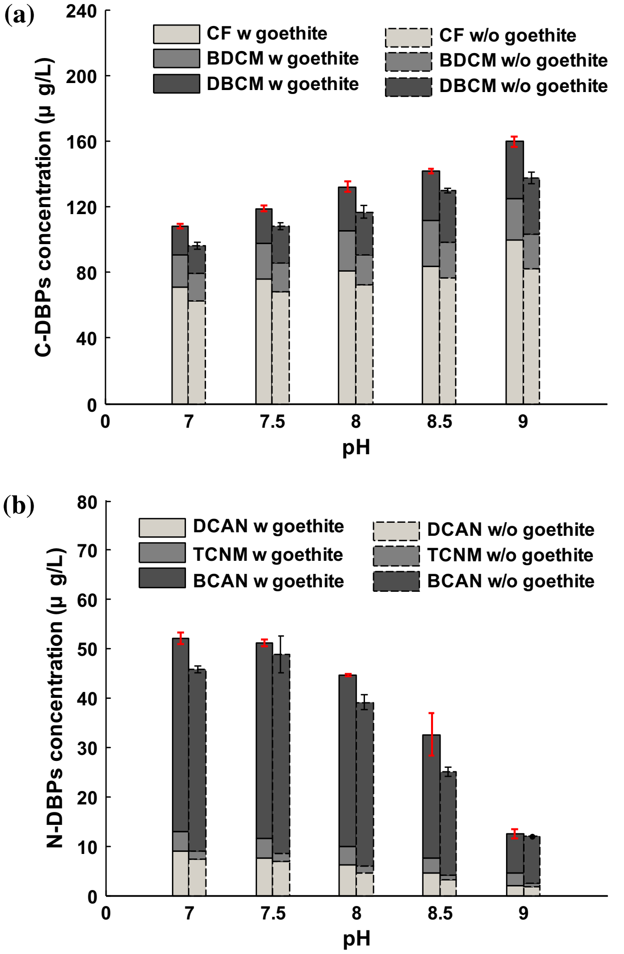

Fig. 2. Effect of pH on the formation of C-DBPs (a) and N-DBPs (b) during chlorination with the presence of goethite. Reaction time = 24 h, temperature = 20 ± 1˚C, goethite concentration = 4 g/L, Cl_2 concentration = 6 mg/L. Error bars represent one standard deviation of duplicate measurements of C-DBPs or N-DBPs.

DBP precursors with smaller molecular weight [21,32]. Moreover, iron oxides are capable of oxidizing NOM in abiotic systems [33–35], which may enhance the chlorinated reactions. In addition, goethite can adsorb and concentrate NOM onto its surface, which can enhance the reactions between NOM and chlorine on the surface of goethite instead of reactions in the aqueous solution [22].

$$2FeOOH \rightarrow Fe_2O_3 + H_2O \quad (1)$$

$$FeOOH + 2H^+ + e^- \leftrightarrow Fe^{2+} + H_2O + OH^- \quad (2)$$

$$Fe^{2+} + HOCl \leftrightarrow Fe^{3+} + \cdot OH + Cl^- \quad (3)$$

Chorine consumption in the presence of goethite was higher than that in the absence of goethite (Fig. 1(c)). The increase in chlorine consumption may be attributed to the reaction between chlorine and ferrous ions on the surface of goethite [21,31,36] and the reactions between chlorine and modified NOM after $^{\cdot}$OH attack [37]. Research comparing aqueous systems to systems containing pipe deposit suggested that chlorine decay is always higher for the pipe deposit systems [38]. NOM is preferentially adsorbed to goethite surface which subsequently results in increasing reactivity with chlorine. This mechanism is expected to have a significant impact on the formation and speciation of disinfection by-products (DBPs) [37].

3.2. Effect of pH on DBP formation

In water distribution systems, pH is an important factor regarding the formation of DBPs as well as corrosion control because hydrogen ion (H^+) is one of the major species that can accept electrons given up by a metal when it corrodes [39]. Because pH in water distribution systems is always maintained at 7–9 [40], the formation of C- and N-DBPs with and without the presence of goethite during chlorination was tested in this pH range, and the results are shown in Fig. 2. As shown in Fig. 2(a) and (b), the concentrations of both C- and N-DBPs increased in the presence of goethite. The concentration of C-DBPs increased with the increase in solution pH, except for BDCM with a maximum at pH 8.5, while the concentration of N-DBPs decreased as pH increased from 7 to 9. Similar dependence of C- and N-DBPs formation on pH during chlorination has been reported by previous researchers [41–43]. According to the study of Reckhow and Singer [43], higher CF formation at higher solution pH is due to the base-catalyzed hydrolysis of CF precursors with the structure of R-CO-CX_3. The stability of HANs depends greatly on their chemical structures and solution pH [44]. A faster hydrolysis of DCAN in basic solution was reported by Glezer et al. [45], which could explain the decrease in N-DBPs as pH increased in our study.

The effect of pH on DBP formation in the presence of goethite can be explained by the distribution of goethite species at different pH values. Based on the pKa values of goethite ($pKa_1 = 5.3$ and $pKa_2 = 8.8$), it can be present as positively charged $\equiv FeOH_2^+$, neutral $\equiv FeOH$, or negatively charged $\equiv FeO^-$ in solutions [23]. The adsorption of organic compounds mainly happens with $\equiv FeOH_2^+$ and $\equiv FeOH$ [46], which can enhance the reactions between NOM and chlorine as discussed in the previous section.

3.3. Effect of chlorine concentration on DBs formation

Formation of C- and N-DBPs after 24-h chlorination with or without the presence of goethite at different chlorine dosages is shown in Fig. 3. In the presence of goethite, the formation of C- and N-DBPs was higher compared to that without the presence of goethite in all cases (Fig. 3(a) and (b)). As shown in Fig. 3(a), the total concentrations of THMs, including CF, BDCM, and DBCM, increased with the increase in chlorine dosage, and the increment became sluggish at $Cl_2 > 8$ mg/L. According to Rebhunt et al. [47] and Shukairy et al. [48], the formation of chlorinated and brominated DBPs was affected by the ratios of HOCl/Br^-, Br^-/NOM, and Br^-/free chlorine. With the increase in Cl_2/Br^- ratio, the concentrations of CF, DBCM, and BDCM increased [49]. As shown in

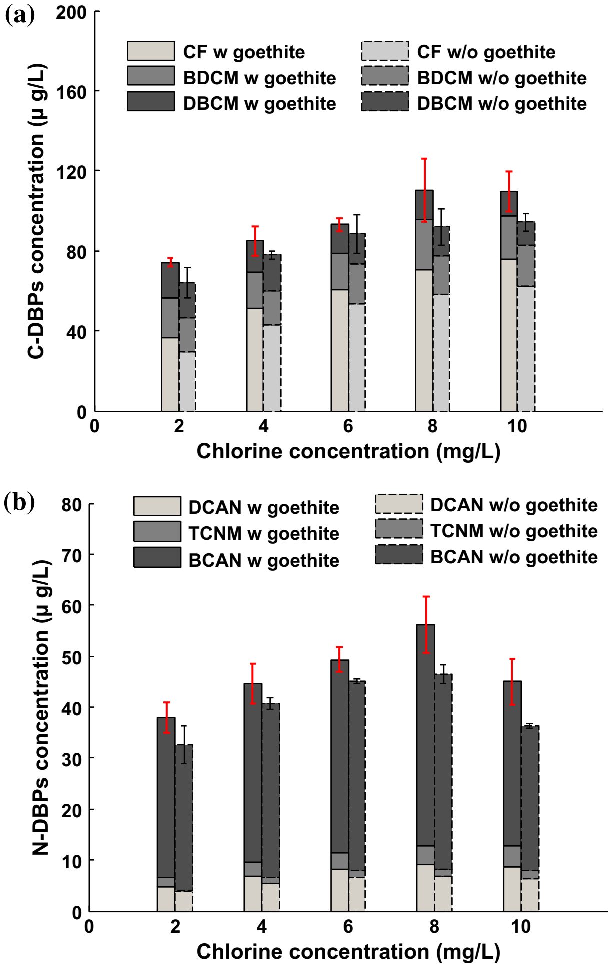

Fig. 3. Effect of chlorine concentration on the formation of C-DBPs (a) and N-DBPs (b) during chlorination with the presence of goethite. Reaction time = 24 h, temperature = 20 ± 1˚C, goethite concentration = 4 g/L, pH 7. Error bars represent the standard deviation of replicate measurements.

Fig. 3(b), DCAN and BCAN concentrations reached a maximum at Cl_2 = 8 mg/L and then decreased as chlorine dosage increased, which could be attributed to fast hydrolysis rates in the presence of excess chorine [50]. The concentration of TCNM increased with increasing chlorine dosage, and the trend was more obvious in the presence of goethite.

3.4. Structure and morphology evaluation of goethite using XRD and SEM

Crystallization of goethite collected from Milli-Q water and chlorinated natural water sample after the filtration process in YDWTP was analyzed using XRD, and the results were shown in Fig. 4. There are fewer peaks detected in goethite collected from chlorinated natural water sample, which may be attributed to the addition of NaOCl, making FeOOH to be more amorphous and/or the break of FeOOH structure by the adsorbed NOM [51], and the adsorbed NOM could become the more acidic fractions [52].

Fig. 5 shows the surface morphology of goethite collected from Milli-Q and chlorinated natural waters observed using SEM. The surface of virgin goethite (Fig. 5(a)) was more compact compared to that collected from chlorinated natural water samples (Fig. 5(b)). Hassan et al. [37] proposed that goethite can adsorb NOM onto its surface and enhance DBP formation by modifying active sites of NOM. In the absence of NOM, the presence of goethite did not significantly affect chlorine decay [36]. Therefore, the porous surface of goethite with some attached blocks could be owing to the adsorption of NOM.

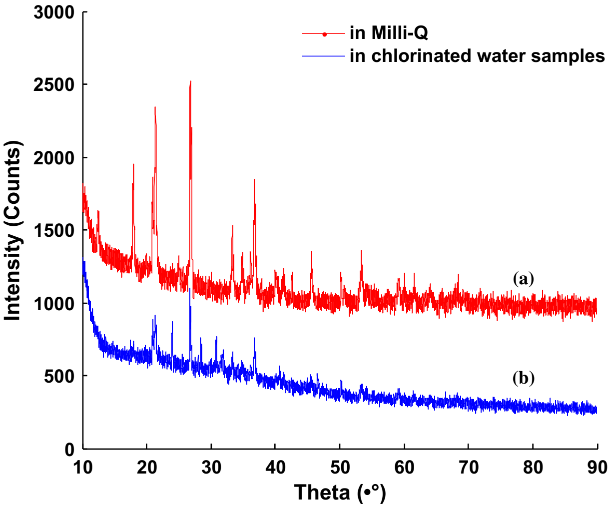

Fig. 4. XRD patterns of goethite collected from Milli-Q water (a) and chlorinated natural water sample (b).

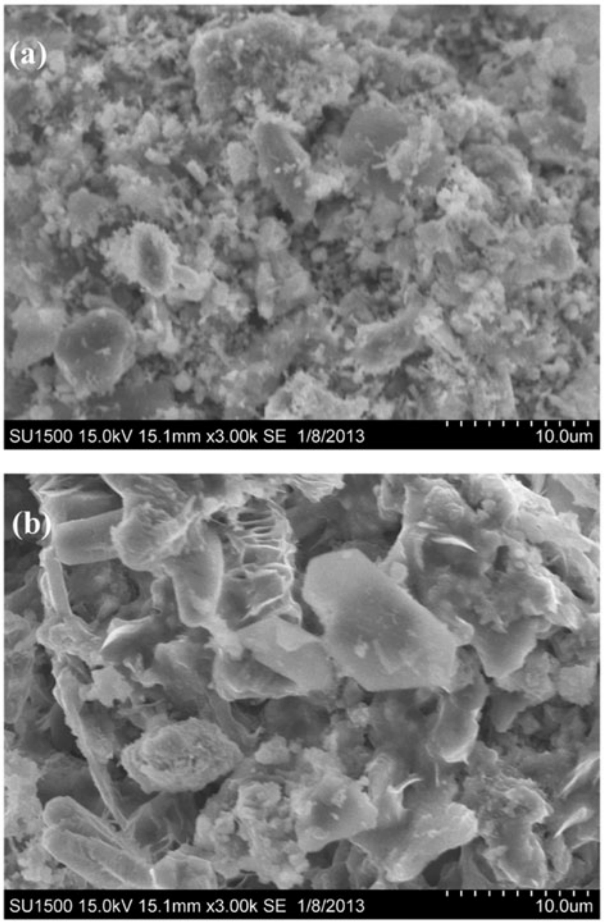

Fig. 5. Surface morphology of goethite in Milli-Q water (a) and collected from chlorinated natural water sample (b) (SEM magnification 3,000×).

3.5. Proposed reaction mechanisms of NOM chlorination in the presence of goethite

C- and N-DBPs formation is significantly enhanced during chlorination in the presence of goethite (Figs. 1 and 2). As discussed in Sections 3.1 and 3.2, the reaction mechanisms are proposed based on NOM adsorption on goethite surface, $^{\cdot}OH$ formation during chlorination in the presence of goethite, and ferric catalysis. The scheme of reaction mechanisms is shown in Fig. 6.

As shown in Fig. 6, the adsorbed NOM on the goethite surface is mainly FeOH with the reactivity of O–H groups, which are important in determining the mobility, reversibility, and the fate of adsorbed species [37]. The intensity of surface reaction is usually stronger than that of aqueous reaction [53]. Moreover, goethite could oxidize HOCl to $^{\cdot}OH$ (Eq. (3)) [36] so as to degrade larger NOM molecules into smaller ones and promote the formation of DBPs. Ferric ions on goethite also could enhance the formation of DBPs [29].

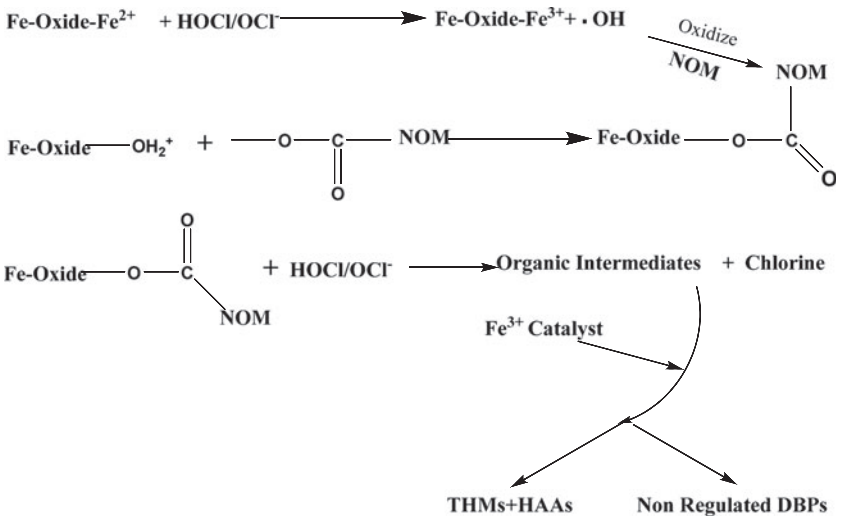

Fig. 6. Proposed reaction mechanisms of NOM chlorination in the presence of goethite.

4. Conclusions

The experimental results indicated that in the presence of goethite, the formation of C- and N-DBPs was enhanced as pH and reaction time increased during chlorination. CF and DBCM formation increased as pH increased from 7 to 9, while BDCM reached a maximum at pH 8.5. N-DBP formation decreased as pH increased. As chlorine concentration increased from 2 to 10 mg/L, the concentration of three C-DBPs and TCNM increased, while the formation of DCAN and BCAN reached a maximum at a chlorine concentration of 8 mg/L. The enhancement of DBP formation in the presence of goethite could be attributed to the generation of ·OH from the reaction between ferrous ion and chlorine, which oxidized NOM into much smaller DBP precursors.

From the XRD results, goethite collected from chlorinated natural water sample showed fewer peaks compared to the virgin goethite. SEM analysis revealed that goethite may adsorb NOM on its surface and modified NOM structure. The adsorbed NOM on the surface of goethite (mainly $FeOH_2^+$ and FeOH) can enhance the reaction between HOCl and NOM compared to that in an aqueous system.

The enhancement of DBP formation in the presence of goethite can be explained by NOM adsorption on goethite surface, ·OH formation during chlorination in the presence of goethite and ferric catalysis, and the scheme of reaction mechanisms that are proposed in this study.

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