



Effect of pH on floc properties and membrane fouling in coagulation – Ultrafiltration process with ferric chloride and polyferric chloride



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HIGHLIGHTS

- Impact of pH on coagulation–ultrafiltration process was investigated.
- Fe (III) species in ferric chloride and polyferric chloride was measured.
- Flocs formed under acidic ranges were larger, weaker and looser.
- Acidic conditions were beneficial to reduce membrane fouling.
- pH dependence was correlated with the Fe (III) species distribution in coagulants.

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ABSTRACT

Impact of pH on coagulation–ultrafiltration (C–UF) process was investigated with respect to coagulation efficiency, floc characteristics and membrane fouling in this study. Ferric chloride (FeCl_3) and polyferric chloride with basicity of 1.0 and 2.2 (denoted as PFC₁₀ and PFC₂₂) were used as coagulants and Fe (III) species in them was measured by a timed complexation spectroscopy method. Floc properties under four pH conditions were evaluated using a laser diffraction particle sizing device. Ultrafiltration experiments were conducted by a dead-end batch unit. The results showed that organic matter removal efficiency was higher under acidic conditions than under other pH conditions and turbidity removal efficiency was higher under alkaline condition. At same pH, FeCl_3 containing higher monomeric and polymeric species (Fe_a and Fe_b) had better organic matter removal and higher turbidity removal efficiency was obtained by coagulants with larger percentage of polymer or colloidal species (Fe_c). Flocs formed under acidic ranges were larger, weaker and looser. At pH 4.0, 7.0 and 9.0, flocs by FeCl_3 were larger and weaker than these by PFC₁₀, followed by PFC₂₂. In case of FeCl_3 and PFC₁₀, acidic pH conditions were helpful to reduce membrane fouling. For PFC₂₂, permeate fluxes were less sensitive to pH variations.

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1. Introduction

Ultrafiltration (UF) technology has become an accepted alternative to conventional treatment in drinking water production. However, UF membrane fouling was one of the most significant issues affecting implementation and performance of UF membranes in drinking water production (Peiris et al., 2013). Many studies (Howe and Clark, 2002; Lee et al., 2006) suggested that natural organic matter (NOM) is the most relevant foulant. And

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humic acid (HA), which was the main component of NOM, was showed to play an important role in membrane fouling (Aoustin et al., 2001). In order to overcome membrane fouling, many approaches were proposed. One aspect was optimization of operation conditions including running modes, rinsing, chemical cleaning and air scouring (Gao et al., 2011). Moreover, some pre-treatments were introduced to reduce membrane fouling during UF process, including coagulation, adsorption, pre-oxidation and pre-filtration, etc. (Huang et al., 2009).

Coagulation/flocculation is widely used as a pretreatment to improve NOM removal and control fouling of UF membranes. As revealed by previous researches (Barbot et al., 2008; Konieczny et al., 2009), the performance of coagulation–ultrafiltration (C–UF) process was closely related to coagulant type, coagulation

condition and resultant floc characteristics. Choo et al. (2007) stated that the degree of fouling reduction during C-UF process highly depended on the type of coagulants used. Waite et al. (1999) concluded that ultrafiltration fouling had close correlation with the size and structure of flocs formed in suspension during C-UF treatment. Moreover, pH of feed water could strongly affect NOM removal and membrane fouling in C-UF hybrid process (Kabsch-Korbutowicz, 2006). Dong et al. (2006) reported that the irreversible fouling of membrane could be decreased at lower pH values in HA treatment by C-UF process. Ruohomäki et al. (1998) also stated that the most serious membrane fouling occurred at neutral pH in humic substances removal by C-UF.

In addition, adjustment of pH is a significant way to improve turbidity and NOM removal in enhanced coagulation process (Gregory and Carlson, 2003). pH could not only affect the characteristics of component in feed water but also the speciation of coagulants used (Yan et al., 2007). As ferric-based coagulants are commonly used in coagulation, the monomer coagulant ferric chloride (FeCl_3) and inorganic polymer coagulant polyferric chloride (PFC) have drawn more and more attention. According to previous studies (Jiang and Graham, 1998; Shi et al., 2011), Fe (III) species in ferric coagulants can be divided into three categories by ferron analysis: Fe_a , monomeric species such as Fe^{3+} , $\text{Fe}(\text{OH})^{2+}$, $\text{Fe}(\text{OH})_2^+$, $\text{Fe}(\text{OH})_3$ (molecule) and $\text{Fe}(\text{OH})_4^-$, a dimer and trimer ($\text{Fe}_2(\text{OH})_2^{4+}$ and $\text{Fe}_3(\text{OH})_4^{5+}$), some small polymers and instantaneous reacted species are usually classified into this category; Fe_b , polymeric species with medium and high molecular mass; and Fe_c , larger polymer or colloidal species, no reaction species. The Fe (III) species could be changed by the pH variation during coagulation, which resulted in different coagulation performance. Gao et al. (2007) stated that variation of water pH could lead to change of Fe (III) species in PFC and polyferric chloride–polydimethylallylammonium chloride (PFC–PDMDAAC), which gave rise to different color removal efficiency under different pH conditions. However, there are few literatures about floc properties and membrane fouling during C-UF process coagulated by ferric coagulants under different pH conditions.

This study focused on the effect of pH on C-UF process in terms of coagulation efficiency, floc properties and membrane fouling. And, Fe (III) species in FeCl_3 and PFC was also evaluated.

2. Methods and materials

2.1. Synthetic feed sample

The preparation of HA stock solution was as follows (Feng et al., 2015): 1.0 g HA (Aladdin, Shanghai, China) and 0.4 g NaOH were dissolved in 800 mL deionized water under continuous stirring and then diluted to 1000 mL with deionized water.

Kaolin was used to simulate suspension in surface water. Kaolin stock solution was prepared by dissolving 5.0 g kaolin (Tianjin, China) in 800 mL deionized water. The clay suspension was diluted to 1 L with deionized water after stirring for 30 min, and then settled for 30 min in a glass measuring cylinder. The top 500 mL was decanted for later use.

A HA-kaolin synthetic water was prepared by adding HA stock solution into tap water to make its concentration 10 mg L. The initial turbidity was adjusted by kaolin stock suspension to 15.0 ± 0.5 NTU. The properties of synthetic water were: turbidity = 15.0 ± 0.5 NTU, $\text{UV}_{254} = 0.315 \pm 0.011 \text{ cm}^{-1}$, $\text{DOC} = 4.671 \pm 0.251 \text{ mg L}$, Zeta potential = $15.4 \pm 0.5 \text{ mV}$ and $\text{pH} = 8.38 \pm 0.02$.

2.2. Coagulants

All reagents used were of analytical grade and deionized water was used to prepare all solutions. The procedures of coagulants preparation were as follows.

FeCl_3 solution: $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ was directly dissolving in deionized water to obtain FeCl_3 solution.

PFC: Firstly, FeCl_3 solution was obtained by dissolving $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in deionized water. Then, Na_2CO_3 solutions with predetermined amounts of Na_2CO_3 powder were gradually titrated into FeCl_3 solution at room temperature to obtain PFC with $\text{OH}^-/\text{Fe}^{3+}$ molar ratios (basicity, B) of 1.0 and 2.2 (denoted as PFC_{10} and PFC_{22}). The mixture was stirred until foam disappeared. Finally, $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ as stabilizer was added into the PFC solution to adjust the molar ratio of P and Fe to 0.08. The concentration of Fe^{3+} was 10 g L in the target PFC solutions.

The Fe (III) species distribution of coagulants was measured by a timed complexation spectroscopy method (Gao et al., 2007; Wei et al., 2009). A brief description of this method was as follows: as Fe in each coagulant reacted with Ferron (8-hydroxy-7-iodoquinoline-5-sulphonic acid) to form metal complex, visible light absorbance was measured as a function of time at a wavelength of 600 nm to quantify the amount of Fe complex. Fe (III) species can be measured according to the reaction time between coagulants and Ferron: reaction within 1 min (Fe_a), reaction over 3 h (Fe_b) and colloidal and no reaction (Fe_c). The Fe (III) species distribution of coagulants was shown in Table 1.

2.3. Jar test

Standard jar tests were performed in a 1.0 L plexiglass beaker using a programmable jar-test apparatus (ZR4-6, Zhongrun Water Industry Technology Development Co. Ltd., China). The solutions were stirred rapidly at 200 rpm for 1.5 min, followed by slow mixing at 40 rpm for 15 min (Dong et al., 2014). Coagulant was dosed after the solution was stirred rapidly for 30 s. Then after 20 min settling, a sample was collected 2.0 cm below the surface for subsequent measurements. Turbidity was measured using a 2100P turbidimeter (Hach, USA) immediately after sample collection. Zeta potential of the supernatant water was measured using a Zetasizer 3000Hsa (Malvern Instruments, UK). The sample was filtered through 0.45 μm fiber membrane before DOC analysis using a Shimadzu TOC-VCPH analyzer and UV_{254} (absorbance at 254 nm) measurement using a TU-1810 UV/VIS spectrophotometer.

2.4. Floc properties

Coagulation experiments were carried out similarly as the jar tests mentioned in Section 2.3. However, after the slow stirring, the suspension was exposed to increased shear forces of 100, 150, 200, 250 and 300 rpm for further 5 min, and then slow stirring at 40 rpm was introduced again for 15 min to allow flocs re-growth. As coagulation proceeded, evolution of particle size was monitored by a laser diffraction instrument (Malvern Mastersizer 2000, Malvern, UK) every 30 s and the corresponding data was automatically recorded. In this study, 50 percentile floc size ($d_{0.5}$) was used to denote floc size.

As the rate at which a floc size decayed under exposure to shear was indicative of floc strength, the relation between the average velocity gradients G in the flocculation and the floc size of the suspension in equilibrium was used by many researchers (Jarvis et al., 2005a; Wang et al., 2009):

$$\lg d = \lg C - \gamma \lg G \quad (1)$$

Table 1
The Fe (III) species distribution of coagulants.

Coagulant	Fe_a (%)	Fe_b (%)	Fe_c (%)
FeCl_3	76.39	21.14	2.47
PFC_{10}	50.84	33.59	15.57
PFC_{22}	15.07	37.75	47.18

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