RESEARCH ARTICLE

Applying chemical sedimentation process in drinking water treatment plant to address the emergent arsenic spills in water sources

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Abstract Arsenic (As) spills occurred more frequently and sometimes polluted water sources in recent years in China. It is as urgent need to develop emergency treatment technologies to address the arsenic threat for large-scale water treatment plants. In response, we developed a chemical sedimentation technology to remove arsenic contaminants for water treatment plants. Bench-scale experiments were conducted to investigate the efficiency of arsenic removal and the influencing factors of the chemical sedimentation treatment process. The influencing factors included the choice and dosage of coagulants, the valence of arsenic and pH value of solution. The As(V) contaminants can be almost completely removed by ferric or alum coagulants. The As(III) contaminants are more recalcitrant to chemical sedimentation, 75% for ferric coagulant and 40% for alum coagulant. The quantitative results of arsenic removal load by different ferric or alum coagulants were presented to help determine the parameters for arsenic treatment technology. The dominant mechanism for arsenic removal is static combination, or adsorption of negative arsenic species onto positive ferric hydroxide or alum hydroxide flocs. The efficiency of this treatment technology has also been demonstrated by a real production test in one water treatment plant with arsenic-rich source water and one emergency response. This technology was verified to be quick to set-up, easy to operate and highly efficient even for high concentration of arsenic.

Keywords Arsenic spill, chemical sedimentation, coagulation, drinking water, emergency treatment

1 Introduction

Arsenic (As) contamination in water is a worldwide problem due to the natural and artificial reasons. Long-term exposure to the drinking water containing high concentration arsenic would increase the risks of cancer, particularly of the skin, lung, bladder, and kidney [1–4]. Many countries and the World Health Organization (WHO) have strengthened the arsenic regulations in drinking water due to its high toxicity. In 1993, the WHO revised the arsenic in its drinking water guideline from 0.05 to 0.01 mg·L^{-1} [5]. The United States and the European Union followed the WHO and set the arsenic standard level at 0.01 mg·L^{-1} [6]. In 2006, the China also tightened its criteria for arsenic concentration in drinking water from 0.05 to 0.01 mg·L^{-1} [7]. The more strict regulations of arsenic increase the difficulty of arsenic removal in water treatment plants, especially for emergency response.

In addition to natural occurrences, industry applications of arsenic also would cause arsenic pollution such as herbicides, insecticides, wood preservatives, alloys, pharmaceuticals, etc [8,9]. Recently, due to the illegal discharge of industrial wastewater, arsenic spills in surface water occurred frequently in China [10,11]. For example, in December 2007, a sulfuric acid plant illegally discharged its wastewater containing a high concentration of arsenic into the Duliu River, Guizhou Province, which caused an arsenic contamination accident [12]. The arsenic-rich water poisoned over one dozen local villagers and threatened the drinking water supply of more than 20,000 people who live in Sandu County at the downstream of Duliu River. Another arsenic spill accident happened in December 2008. A chemical plant discharged its arsenic-rich washing water of phosphate tail gas into the Picang flood channel, Shandong province, China [13].

There are several technologies have been developed to remove arsenic from water, such as enhanced coagulation-sedimentation, adsorption, anion exchange, reverse osmosis, etc [14–18]. However, adsorption, anion exchange and reverse osmosis are applied mostly for arsenic removal in small size water treatment facilities. They are not applicable in large scale surface water treatment plants, especially under emergency response situations due to long preparation time. The enhanced coagulation-sedimentation, as also named as the chemical sedimentation process, is more favorable in the emergency response situations. Since the coagulation-filtration process is part of the conventional treatment process, it can be quickly implemented and easily operated in drinking water treatment plants.

In the previous studies, the arsenic valence, coagulant type and dosage, pH value, and water composition were found to be important factors for arsenic removal by the chemical sedimentation process. The previous studies have noted that the As(V) contaminants are more easily removed than As(III) contaminants under the same coagulation condition. Thus, the pre-oxidation will transform the As(III) into As(V) and favorite the total arsenic removal. The mechanism of arsenic removal by coagulation is quite complicate and still not very clear. It is probably a combination of precipitation, adsorption, exchange, co-precipitation effect.

However, the previous reports on the enhanced coagulation-filtration for arsenic removal are mainly conducted on bench scale. The removal efficiency varies with water quality and treatment process in different reports. The difference of the coagulants and the pH value also increases the uncertainty of arsenic removal [19–22]. Drinking water treatment plants need more detail to guide the chemical sedimentation operations. Moreover, the majority of previous studies focused on the treatment of arsenic contaminated water with slightly excess of the standard [23]. There is little report for drinking water treatment plants to address the arsenic-rich water with several to dozens of times of criteria which could probably occur in the arsenic spill accidents. It is in urgent demand to study the arsenic removal by chemical sedimentation process in water treatment process to prevent the public health from the arsenic spill accidents.

Therefore, the objectives of this study include: 1) to better understand the chemical sedimentation processes and the impact factors, such as the kinds and dosage of coagulants, the influence of arsenic valence and pH value; 2) to verify the applicability of chemical sedimentation technology in the emergency treatment responses for arsenic spills, especially with high contaminant concentration. Both bench scale study and production tests are included in this paper. These information will help the water treatment plants to address the arsenic contaminations or accidents.

2 Methods and materials

2.1 Materials

Arsenic exists in different states in the environment, such as As(V), As(III), As(0) and As(-III) [6]. Arsenic states depend on the pH and the redox potential of the solution. The most common arsenic states in aqueous solution are As(III) and As(V). Arsenic mainly exists as As(V) in the aerobic environment and As(III) exists in the anoxic environment, such as ground water or sediment of deep lake [1].

To study the influence the arsenic valence on the removal after treatment, both As(V) and As(III) were tested in this study. The As(V) contaminant was prepared from $Na_2HAsO_4 \cdot 7H_2O$ solution. The As(III) was prepared by solving As_2O_3 with 2% HNO_3 to get the $1.0 \, g \cdot L^{-1}$ of arsenite solution. These arsenic chemicals were bought from Sigma-Aldrich (> 98%, USA).

The coagulants, including ferric chloride ($FeCl_3 \cdot 6H_2O$, 97%, Sigma-Aldrich, USA), polymeric ferric sulfate (PFS, 18% Fe, Tianjin Guangfu Fine Chemicals Research Institute, China), aluminum sulfate ($Al_2(SO_4)_3$, 97%, Sigma-Aldrich, USA) and poly-aluminum chloride (PACl, 28% Al_2O_3, Tianjin Guangfu Fine Chemicals Research Institute, China), were applied in this study.

2.2 Bench-scale test

In the bench-scale test, the initial concentration of arsenic was set as $0.1 \, mg \cdot L^{-1}$ or $0.05 \, mg \cdot L^{-1}$, which is the 10 times or 5 times of the criteria in the drinking water quality standards of China [7]. The coagulation experiments were conducted by a jar tester (ZR4-6, Zhongrun Water Industry Technology Development Co. Ltd., China). The coagulation-sedimentation process was simulated as a fast mixing ($300 \, r \cdot min^{-1}$ for 1 min), flocculation process ($60 \, r \cdot min^{-1}$ for 5 min, $30 \, r \cdot min^{-1}$ for 10 min) and settling for 30 min. Then, the water samples were filtered by paper filters to simulate the filtration process in water treatment plants. The first 100 mL filtered water was discarded.

To determine the parameters of coagulation, the coagulant concentrations were set as $3 \, mg \cdot L^{-1}$, $5 \, mg \cdot L^{-1}$ and $10 \, mg \cdot L^{-1}$ of iron or aluminum separately. The initial pH values of solutions were set as 7.0, 7.5, 8.0, 8.5, 9.0 and 9.5, and adjusted by hydrochloric acid (volume ratio of 1:9) and sodium hydroxide ($1 \, mol \cdot L^{-1}$).

As(III) and As(V) were used to determine the impact of the arsenic valence on their removal. Different concentrations of chlorine were also added to study the impact of the oxidation on removal of As(III). After 10 min chlorination, $10 \, mg \cdot L^{-1}$ of coagulant was added in the solution. Then, the conventional coagulation, sedimentation and filtration treatment was simulated and the arsenic concentration in filtrate was measured.

2.3   Analytical methods

Arsenic concentration were detected by hydride generation atomic fluorescence spectrometry (AFS930, Jitian Corporation, China) according to the Standard Examination Methods for Drinking Water of China [24].

A pH electrode (Orion 4 STAR, Thermo Scientific, USA) was used for pH analyzed. Other water quality indexes were measured according to the Standard Examination Methods for Drinking Water in water treatment plants [24]. The turbidity was measured by a portable turbidity meter (2100P, Hach, USA).

3   Results and discussion

3.1   Influencing factors on arsenic removal

3.1.1   Arsenic valence

The arsenic valence will influence the removal of total arsenic greatly. The pre-oxidation will transform the recalcitrant As(III) into As(V) and retrieve the removal of total arsenic, as shown in Fig. 1.
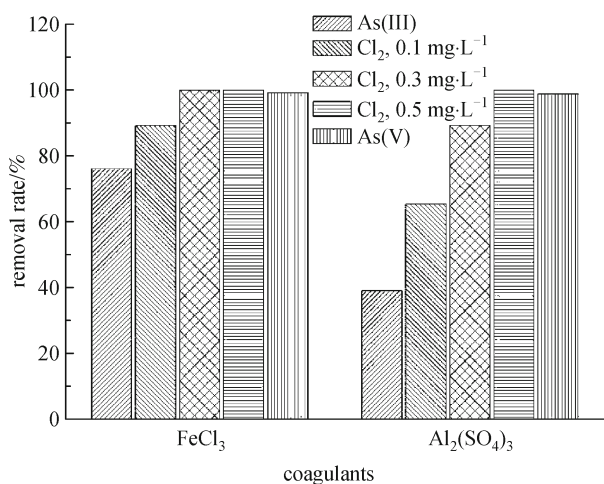
Fig. 1   The effects of the arsenic valence and the oxidant dosages on arsenic removal in the coagulation treatment process (Raw water parameters: turbidity = 38 NTU; alkalinity = 122 mg·L^{-1}; hardness = 172 mg·L^{-1}; pH = 7.88. The coagulant doses are 10 mg·L^{-1} (Fe or Al). Total As concentration = 0.1 mg·L^{-1})

As demonstrated in Fig. 1, both ferric and aluminum coagulants can remove As(V) almost completely, but As(III) is much more recalcitrant to sedimentation process, especially for the alum coagulation. The removal of As(III) by ferric coagulant reduced to 76% and the number for aluminum was only 39% with the same mass dosage of 10 mg·L^{-1}. The removal of As(III) will be improved with pre-chlorination because As(III) can be quickly oxidized into

As(V) by chlorine [25]. If the source water contains a high concentration of As(III), pre-oxidation would be necessary to improve the total arsenic removal.

3.1.2   Coagulants and dosages for Arsenic removal

As revealed in Fig. 1, As(III) was only partially removed by coagulation process without pre-oxidation. Thus, the emphasis was laid on the influence of coagulant concentration and the pH of solution on As(III) removal.

As Fig. 2 demonstrated, increasing the ferric or alum coagulant dosage would improve the arsenic contaminants removal. The ferric coagulants could remove As(III) more efficiently than aluminum coagulants, 60%–80% vs. 30%–50% with the same mass dosage. The arsenic removal capacities onto aluminum compared with ferric was the same as previous studies [26]. This difference could be explained by that ferric has a higher Lewis acid (electron pair acceptor) strength or affinity for arsenic anions, which are Lewis basic (electron pair donors), than alum [27,28].

The coagulants with same metal had the similar arsenic removal. There is no much difference on arsenic removal between FeCl$_3$ and PFS or between Al$_2$(SO$_4$)$_3$ and PACl. Increasing coagulants dosage from 3 mg·L^{-1} to 10 mg·L^{-1}, the As(III) removal efficiency by ferric coagulants increased from 60% to 80% and that by alum coagulants increased from 30% to 50%. Therefore, it is hardly to satisfy the drinking water criterion even with much high coagulant dosage in the occurrence of high initial As(III) concentration. It is highly recommended to add chlorine or other oxidants to oxidize the As(III) into As(V) before coagulation treatment.

Further comparisons of arsenic removal load were made between different valence of arsenic and different
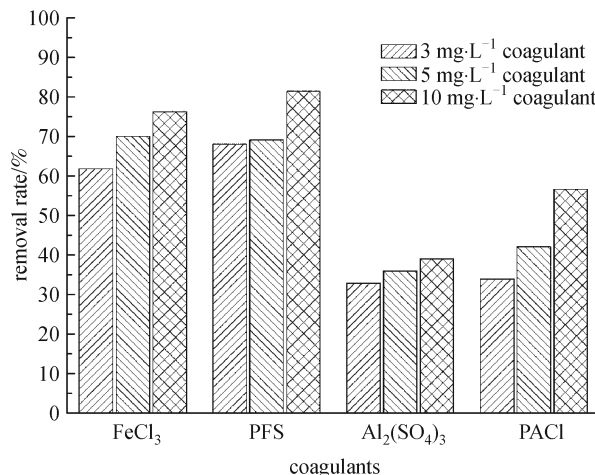
Fig. 2   The removal efficiency of As(III) by different coagulants at different concentration (Raw water parameters: turbidity = 21 NTU; alkalinity = 124 mg·L^{-1}; hardness = 174 mg·L^{-1}; pH = 7.77. Total As concentration = 0.1 mg·L^{-1})

coagulants, as shown in Fig. 3. The curves of normalized arsenic removal load of different coagulants showed good linear relationship with the arsenic concentration at equilibrium, whether As(III) or As(V). The coefficients of these curves, i.e. r^2 are as high as over 0.97 except one.

It is interesting that PACl and $FeCl_3$ coagulants have almost the same As(V) removal load, which indicate the As(V) removal is independent of coagulants. Meanwhile, the As(III) removal varies with coagulants: ferric coagulants have 6–7 times higher As(III) removal load than alum coagulants with the same equilibrium concentration. The two ferric coagulants, i.e. $FeCl_3$ and PFS, have almost the same As(III) removal load curve and the same slope rate, which indicate the two ferric coagulants might hydrolyzed into the same ferric species that react with As(III) species and then precipitate. However, the slope rate of PACl and that of $Al_2(SO_4)_3$ is quite different, which implies the two alum coagulants have quite different reactive species to remove As(III).
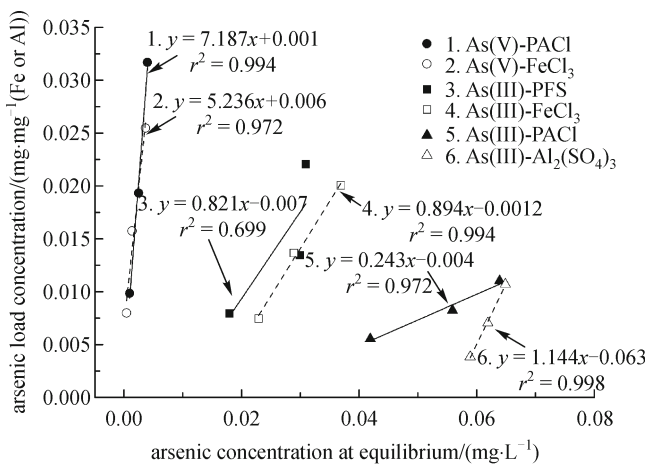
Fig. 3 The comparison of As removal load by different coagulants at equilibrium

Figure 3 also presents important information for arsenic removal practice. We can determine the coagulants for arsenic removal and calculate the needed dosage. Under the equilibrium concentration of $0.01\ mg \cdot L^{-1}$, i.e., the maximum concentration level (MCL) of arsenic in drinking water standard in China, USA or WHO, the load of As(V) of coagulants is $0.073\ mg\ As \cdot mg^{-1}$ Al or $0.058\ mg\ As \cdot mg^{-1}$ Fe. Thus, the minimum dosage for As(V) removal is only $0.13\ mg \cdot L^{-1}$ for PACl or $0.17\ mg \cdot L^{-1}$ for $FeCl_3$. In other words, $10\ mg \cdot L^{-1}$ of alum or ferric coagulants could theoretically handle the initial concentration of $0.73\ mg \cdot L^{-1}$ or $0.58\ mg \cdot L^{-1}$ of arsenic and satisfy the drinking water standard. This calculation was proven by our experiment that $10\ mg \cdot L^{-1}$ of $FeCl_3$ could remove high As(V) concentration of $0.5\ mg \cdot L^{-1}$ or $1.0\ mg \cdot L^{-1}$ into only $0.02\ mg \cdot L^{-1}$ or $0.05\ mg \cdot L^{-1}$.

Similarly, we can get the needed dosage for As(III)

removal by PFS is $8.26\ mg \cdot L^{-1}$, about 50 times higher of As(V) treatment. However, this calculation will give minus value for As(III) removal by alum coagulants, which indicates the alum coagulants cannot handle the As(III) contaminants at this level.

3.1.3 pH value for As(III) removal

The pH value also plays an important role in the coagulation process. It influences not only on the coagulation process, but also on the arsenic speciation in the solution.

As shown in Fig. 4, the pH of effluent significantly influence the removal of As(III). The As(III) removal efficiency would rise from 40% to 60% as pH increased from 7 to 9 by ferric coagulants and the efficiency by aluminum coagulations also increased from 20% to 40% in the condition without competitive.
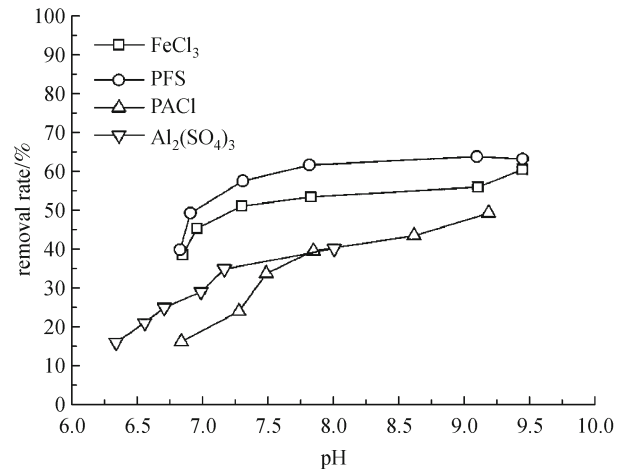
Fig. 4 The effect of pH on the removal of As(III) by different coagulant (Raw water parameters: turbidity = 0.04 NTU; alkalinity = $44.75\ mg \cdot L^{-1}$; hardness = $125\ mg \cdot L^{-1}$. The coagulant doses are $5\ mg \cdot L^{-1}$ (Fe or Al). Total As concentration = $0.051\ mg \cdot L^{-1}$)

The pKa values of H_3AsO_3 are 9.4, 12.2 and 13.6, respectively. The ratios of anionic $H_2AsO_3^-$ in As(III) species are 5% at pH 8 and 40% at pH 9. This ratio matched the As(III) removal by alum coagulants, which indicated that electrostatic adsorption is more likely the responsible mechanism during the coagulation at high pH value. However, for the As(III) removal in ambient pH 6–8 or As(III) removal by ferric coagulants, other mechanism such as co-precipitation could play more important role.

3.1.4 Mechanism of arsenic removal by coagulation process

According to the above results, the arsenic removal was influenced greatly by the arsenic valence, coagulants and pH value. The As(V) contaminants can be completely

removed by ferric or alum coagulants and its removal is independent of coagulants. The As(III) contaminants are more recalcitrant to coagulation and its removal varies with coagulants greatly. This information gives some clues to find the mechanism for arsenic removal.

It is obvious that As(V) and As(III) are removed via different pathways. Previous studies summarized that the As(V) contaminants were removed by coagulation and sedimentation via three possible mechanisms [29].

The first hypothesis is the chemical precipitation mechanism, where arsenic combines with metal ions to form the non-soluble species, such as $FeAsO_4$ ($K_{sp} = 5.7 \times 10^{-21}$) or $AlAsO_4$ ($K_{sp} = 1.6 \times 10^{-16}$), and then precipitate. However, as far as the low arsenic concentration of criteria, i.e., 0.01 mg·L^{-1} to be concerned, it is impossible to satisfy the K_{sp} value of arsenate non-soluble species with very little free ferric or aluminum ions in the flocs at ambient pH conditions.

The second mechanism is co-precipitation, where soluble arsenic species were incorporated into a growing ferric or alum hydroxide flocs via inclusion, occlusion, or adsorption during the coagulation process. Co-precipitation appeared to be the crucial mechanism for unionized arsenic species removal by chemical sedimentation process according to the previous studies [30].

The third mechanism is static adsorption, where arsenic can be adsorbed onto the metal hydroxide by the surface charge. Ferric coagulants can form polymerization complexes or multi-component polymers such as single-core components ($Fe(OH)_2^+$、 $Fe(OH)^{2+}$) and multi-core components ($Fe_2(OH)_2^{4+}$, $Fe_3(OH)_2^{5+}$, etc) in ambient pH conditions. Meanwhile, As(V) exists as hydrogen arsenate ($HAsO_4^{2-}$) in ambient pH according to its pKa value, which can be easily adsorbed onto the cationic flocs. Similarly, the aluminum coagulant can also form the cationic hydroxide flocs and remove As(V) effectively by static combination and adsorption [26,28,31,32].

For As(III), the much lower removal when compared with As(V) could be explained by the different charge of respective species and the different combination ability with either ferric or alum ions. The majority of As(III) species exist as H_3AsO_3 in the pH range of 6–8, and can hardly be removed by electrostatic interaction. Thus, As(III) can be more likely removed by the co-precipitation and physical adsorption mechanism.

Moreover, the difference of As(III) by ferric and alum coagulants could be explained by two properties: 1) the diameter of H_3AsO_3 or $As(OH)_3$ is closer to that of $Fe(OH)_3$ rather than that of $Al(OH)_3$, which also increase the possibility of co-precipitation products of $As(OH)_3$ and $Fe(OH)_3$; 2) The ferric and aluminum ions also have a different Lewis acid (electron acceptor)strength or affinity for the As(III)species, which are Lewis bases (electron donors) [28].

The two ferric coagulants, i.e., $FeCl_3$ and PFS, have almost the same As(III) removal ability, which indicate the two ferric coagulants might hydrolyzed into the same ferric species, react with As(III) species and then precipitate. However, the As(III) removal of PACl and that of $Al_2(SO_4)_3$ is quite different, which implies the two alum coagulants have quite different reactive species to remove As(III).

In all, the possible mechanism of arsenic removal during coagulation process is the combination of co-precipitation with metal hydroxides and the adsorption onto metal oxides, which correspond to the formation of surface complexes between arsenic species and the solid metal hydroxide surface sites. Further investigation is needed to better understand the mechanism and the contribution of each pathway.

3.2 Case studies for arsenic removal in surface water treatment plants

#### 3.2.1 The treatment of arsenic-rich source water

One water treatment plant in southern China suffered high arsenic concentration in source water. The initial arsenic concentration of natural spring water ranged from 0.015 to 0.025 mg·L^{-1}, which meet the environmental quality standards for surface water but over the drinking water quality standards. The majority of arsenic exist as As(III) due to the geological reason.

To address the arsenic-rich water, the chemical sedimentation process was applied. As shown in Fig. 5, 0.5 mg·L^{-1} of free chlorine was added and the alum coagulant was replaced by ferric chloride (imaginary line) according to the bench-scale experiment results. The pH value would fall from 7.8 to 7.5 as increasing the ferric coagulant dose from 0.5mg·L^{-1} to 3.0 mg·L^{-1}. It was not
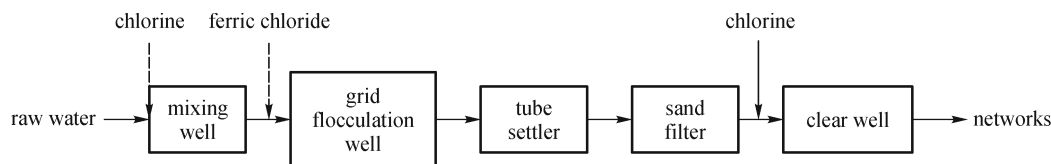
Fig. 5 the modification flowchart of water treatment plant to remove arsenic (The pH and turbidity in raw water were 7.8–8.1 and 0.7–1.1 NTU, respectively. The initial Arsenic concentration was 0.015–0.025 mg·L^{-1})

necessary to adjust pH value since it was still in proper pH range for arsenic removal by coagulation. Free chlorine solution was added in the mixing well to transform the possibly As(III) species into As(V). The performance of chemical sedimentation was illustrated in Fig. 6.
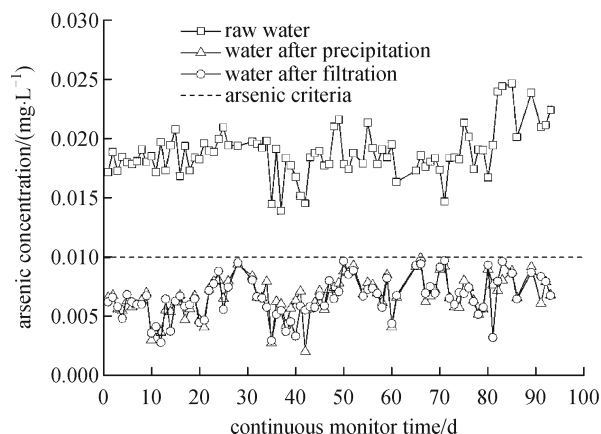
Fig. 6 The arsenic concentration in the effluent in the tracking study (The pH and turbidity in raw water were 7.8–8.1 and 0.7–1.1 NTU. The coagulant doses are 10 mg·L^{-1})

The modified treatment process was very efficiency for water treatment plants with arsenic containing raw water. As shown in Fig. 6, the arsenic concentration in the treated water met the drinking water standards in a three-month tracking study.

3.2.2 The application in an arsenic spill accident

In December 2007, an arsenic spill accident occurred in the Dushan County, Guizhou province. A large amount of arsenic containing wastewater was discharged into the Duliu River. This accident poisoned over a dozen local villagers and threatened the drinking water supply of more than 20,000 people who live downstream of Duliu River in Sandu County. The detected maximum arsenic concentration in the Duliu River was 2.5 mg·L^{-1}, which is 50 times of the environmental quality standards for surface water and 250 times of the drinking water quality standards. The water treatment plant of the Sandu County had to stop taking water from the Duliu River after the accident occurred.

After the accident occurred, response to the requirement of local water treatment plants, our team helped to develop the emergency treatment process for arsenic removal in two days. The modification of water treatment process included: 1) Adding chlorine dioxide blend in the mixing well to transform the possibly existed As(III) species into As(V). The onsite generator of chlorine dioxide used chlorate as the raw material and the products are actually the mixture of chlorine dioxide (over 65%) and free

chlorine (about 35%). 2) Replacing the poly aluminum chloride coagulant by poly ferric sulfate. The iron coagulant dosage was set as 10 mg·L^{-1} at the first days and reduced gradually with the decrease of influent arsenic concentration. 3) Modifying the operation of sedimentation and filtration. The sludge of sedimentation tank was discharged and the filter was backwashed more frequently. The level of turbidity in the filter effluent was set as no more than 0.3 NTU to avoid the leakage of arsenic contained particles.

This arsenic treatment process was established in two days and performed well to satisfy the drinking water standard. The local Health Inspection Agency, who is in charge of the water safety surveillance, announced the success of this emergency treatment process after monitored the treated water quality. The local government announced to resume the water supply in another two days. The arsenic concentration in the treatment processes were demonstrated in Fig. 7.
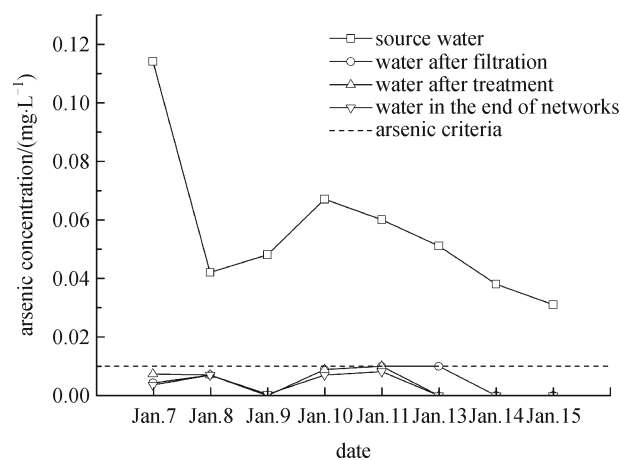
Fig. 7 The arsenic concentration in the treatment process

This case study testified that the process of pre-oxidation and ferric coagulation process can effectively remove arsenic in the arsenic spill accidents. The treatment process would response to the severe arsenic contamination based on the conventional treatment process. The emergency treatment process can be quickly implemented and easily operated.

The financial cost of this application is also very reasonable. The cost of coagulant, poly ferric sulfate (PFS, 18%Fe), was 0.0556 CNY for cubic meter water, if coagulant was dose as 5 mg·L^{-1} and the price of coagulant was 2,000 CNY·t^{-1}. Pre-oxidant, chlorine dioxide mixture, was 0.0015 CNY for cubic meter water, if the pre-oxidation was 0.5 mg·L^{-1} and the oxide price was 3,000 CNY·t^{-1}. The total increase of operation cost was no more than 0.057 CNY for cubic meter water, which is very acceptable by the local water company.

4   Conclusions

The emergency treatment technology of ferric coagulation with the aid of pre-oxidation was developed to effectively address the arsenic contaminants in surface drinking water treatment plants.

The arsenic removal was influenced greatly by the arsenic valence, coagulants and pH value. The dosage of ferric or alum coagulants has small effect on arsenic removal. The As(V) contaminants can be completely removed by ferric or alum coagulants. The As(III) contaminants are more recalcitrant to coagulation, 75% for ferric coagulant and 40% for alum coagulant. The quantitative results of arsenic removal load by different ferric or alum coagulants were presented to help determine the parameters for arsenic treatment technology. If the raw water containing a high concentration of As(III), the pre-oxidation will be necessary to improve arsenic removal. Ferric coagulants can remove arsenic more efficiently than aluminum coagulants. The possible mechanism of arsenic removal during coagulation process is the combination of co-precipitation with metal hydroxides and the adsorption onto metal oxides, which correspond to the formation of surface complexes between soluble arsenic species and the solid metal hydroxide surface sites.

The applications in one production operation and one real emergency response have testified the high efficiency and applicability of this arsenic removal technology in drinking water treatment plants.

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