

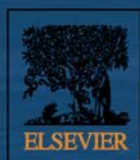
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# Sulfide elimination by intermittent nitrate dosing in sewer sediments

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## ABSTRACT

The formation of hydrogen sulfide in biofilms and sediments in sewer systems can cause severe pipe corruptions and health hazards, and requires expensive programs for its prevention. The aim of this study is to propose a new control strategy and the optimal condition for sulfide elimination by intermittent nitrate dosing in sewer sediments. The study was carried out based on lab-scale experiments and batch tests using real sewer sediments. The intermittent nitrate dosing mode and the optimal control condition were investigated. The results indicated that the sulfide-intermittent-elimination strategy by nitrate dosing is advantageous for controlling sulfide accumulation in sewer sediment. The oxidation–reduction potential is a sensitive indicator parameter that can reflect the control effect and the minimum N/S (nitrate/sulfide) ratio with slight excess nitrate is necessary for optimal conditions of efficient sulfide control with lower carbon source loss. The optimal control condition is feasible for the sulfide elimination in sewer systems.

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## Introduction

The production and the emission of hydrogen sulfide by anaerobic microorganisms in sewer systems are a well-documented problem. The emission of hydrogen sulfide produced in anaerobic zones within the biofilm and in sediments that cover the submerged sewer wall to the sewer atmosphere induces the biological production of sulfuric acid, which causes serious corrosion and health problems (Pomeroy, 1990; Hvitved, 2002; Aysen, 2003). In severe cases, hydrogen sulfide can cause a corrosion rate of around 5 mm/year in a concrete surface (Roberts et al., 2002). In Los Angeles, about 10% of the sewer pipes are prone to sulfide corrosion, and the rehabilitation costs of these corroded pipelines are estimated at \$400 million (Sydney et al., 1996). These problems further cause a high working load and expensive rehabilitation and maintenance costs of sewer systems (Sydney et al., 1996; Vincke, 2002).

The control of the production and emission of hydrogen sulfide, which are the leading causes of pipe corrosion and sewer odor, was investigated to solve this biogenic problem. Studies on different control methods, such as injecting oxygen or adding nitrate/nitrite, iron salts, or H<sub>2</sub>O<sub>2</sub>, have been carried out (Zhang et al., 2008). These studies generally involve (1) the prevention of anaerobic conditions by the addition of oxidants (e.g., nitrate, nitrite, and oxygen), (2) the precipitation of formed sulfide with metal salts (e.g., iron, zinc, lead), (3) the elimination of sulfate reduction bacteria (SRB) populations with biocides (e.g., ozone, hydrogen peroxide, and chlorine), (4) increasing the pH by alkali dosing and minimizing sulfide transfer from liquid to gas phase, and (5) improving sewer design (Zhang et al., 2008; Boon et al., 1998; Hobson and Yang, 2000; Gardner and Stewart, 2002; Charron et al., 2004; Gutierrez et al., 2008). For these methods to be effective, chemical or biological continuous dosing by flow-paced and profiled dosing rates was mostly adopted through trial and error methods (Ramon et al., 2011). Thus, the costs of all of these methods are high (0.19–7.2 €/kg S removal) (Zhang et al., 2008). The industry must find a different chemical dosing strategy. And a new chemical dosing method that takes into account the long persistent inhibition effect, such as those of free nitrous acid, was investigated (Ramon et al., 2011; Jiang et al., 2011;

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Jiang et al., 2013a; Jiang et al., 2013b). Thus, a feasible, cost-effective, and efficient control strategy and the optimal conditions of different control methods must be established to rehabilitate and maintain sewer systems.

Control methods such as nitrate dosing with both chemical and biological effects have been favored and are widely used for controlling sulfide formation in sewer networks (Hvitved, 2002; Myhr et al., 2002). The effect of nitrate dosing on sulfide control in laboratory sewer has been mentioned in previous studies (Einarsen et al., 2000; Rodriguez et al., 2005). The sulfur transformation pathways by using important intermediate  $S^0$  in anoxic and anaerobic conditions was reported in the previous studies (Jiang et al., 2009). The influence of nitrate concentration and exposure time on the inhibition levels of SRB and *Methanogenic Archaea* was studied, both in laboratory simulated biofilm reactors of sewers (Jiang et al., 2013a). The effect of nitrate dosing on SRB community change and on nitrate denitrification-sulfide oxidation bacteria (NR-SOB) simulations in biofilms was investigated in previous studies by using laboratory simulated sewer biofilm reactor (Juan et al., 2007; Mohanakrishnan et al., 2009). Furthermore, continuous nitrate dosing at a concentration range of 10 to 40 mg N/L was shown to reduce the sulfide concentration to 0.2–3 mg S/L in main sewers with lengths from 2.4 to 5 km (Bentzen et al., 1995; Saracevic et al., 2006).

However, in practice the build-up of hydrogen sulfide produced in the sediment environment of the gravity sewer system is significant when the sediment was accumulated for a long-term operation of the sewer pipe, especially in the absence of the maintenance and at low flow condition. Moreover, complex carbon, nitrogen, and sulfur cycles simultaneously occur with nitrate dosing in the sewer. Thus, the optimal cost-effective and efficient conditions for sulfide control must include the minimum nitrate dosing amount, the maximum sulfide removal rate, and the minimum carbon source loss in sewer sediments. The minimized loss of organic carbon source will decrease the consumption of easy biodegradable carbon source in wastewater during the transfer process and provide higher biodegradable carbon source for denitrification process in wastewater treatment plants. Although the effectiveness of sulfide control by nitrate dosing has been investigated in many studies, most of which were carried out by using laboratory cultures in biofilm conditions, but the overall demonstration of real sewer sediment conditions and carbon source loss needs to pay more attention. Furthermore, sulfide accumulation concentration and flow dynamics of gas phase and liquid phase have a dynamic behavior, and the dynamics are different and complex in real sewer systems. The optimal control condition for sulfide elimination in the sewer sediment needs to be developed.

This study aims to develop a new, optimal, and effective control strategy and to determine the optimal control condition by intermittent nitrate dosing for sulfide elimination in sewer sediments. The study was carried out by lab-scale experiments with real sewer sediments and by using real domestic wastewater from sewers. Different dynamic intermittent nitrate dosing modes and optimal dosing conditions were investigated. The effects of efficient sulfide control, minimal nitrate dosing, and low carbon source loss were both considered.

## 1. Material and methods

### 1.1. Lab-scale sewer reactors and operation conditions

The reactors were made of Perspex and designed in the laboratory to simulate real sewer conditions (Fig. 1). The laboratory reactor system consisted of two parallel lines, with one being named as the implement control reactor (ICR) that conducted different following nitrate dosing strategies (listed as

ICR1 and ICR2), another was named as the without control reactor (WCR) and served as the background reference (listed correspondingly as WCR1 and WCR2) during experiments. Each reactor had a volume of 5 L with a diameter of 150 mm and a height of 300 mm. Plastic cylinder vessels with a diameter of 100 mm and a height of 80 mm were fixed on three plastic rods inside each reactor as sediment carriers. The sediments were collected from the downstream of full-scale sewer pipes that contain abundant microbial community to simulate the actual sediment environment of sewer systems. The reactor operated for several weeks to reestablish the original configuration of sediment environment of real sewers. There is a head space in the reactor. The reactors were completely covered with aluminum foil to avoid exposing the sewage and sediment to light. Each reactor had a separate sewage feed, discharge, and control as the experiment schemes.

Domestic wastewater was collected from the end of the drainage network near the wastewater treatment plant and was used as the feed. The sewage had weekly variations in sulfate, volatile fatty acids (VFAs), and chemical oxygen demand (COD) concentration. The typical physicochemical parameters of the fresh sewage are: sulfide  $0.20 \pm 0.01$  mg-S/L, sulfate  $65\text{--}80$  mg- $SO_4^{2-}$ /L, VFA  $10.8 \pm 0.5$  mg-COD/L, total COD  $210 \pm 5$  mg-COD/L and soluble COD  $154 \pm 5$  mg-COD/L, ammonia  $45.0 \pm 0.5$  mg-N/L, pH 7.15–7.60, and oxidation-reduction potential (ORP) from  $-70$  to  $-100$  mV. Nitrate was not present in the fresh sewage. Sulfite and thiosulfate were presented in negligible amounts ( $<1$  mg-S/L). The sewage was stored at  $4^\circ\text{C}$  and heated up to  $20^\circ\text{C}$  (temperature of experiment) before it was pumped into the reactors. The system was intermittently fed with sewage through a peristaltic pump in the initial phase of the experiment. Every feed transferred 4.5 L of sewage into the reactor system. The nitrate solution was dosed into ICR to obtain final nitrate concentrations of 10 and 30 mg-N/L. The sample was taken once every 8 hr with the experiment tests. The rate of nitrate ( $r_{NO_3}$ ) and sulfate reduction ( $r_{sulfate}$ ), the consumption rate of COD and VFA ( $r_{COD}$ ,  $r_{VFA}$ ), and the rate of sulfide oxidation ( $r_{sulfide}$ ) were tested during the experiment. A similar hydraulic condition with the real sewer system was obtained by the magnetic stirrers (200 r/min) to simulate the sediment environment of the real sewer system. There is no obvious biofilm with rich sulfate reducing bacteria growth attached in the sewer inside wall because of the periodically cleaning during the experiments.

### 1.2. Design of nitrate dosing schemes

Two strategies of intermittent nitrate dosing mode were developed. The first strategy involves the use of sulfide full elimination (SFE). The nitrate (30 mg-N/L) was dosed into the reactor at once when the sulfide disappeared in bulk water. The second strategy involves the use of sulfide intermittent elimination (SIE). The nitrate (10 mg-N/L) was dosed into reaction when the sulfide accumulated and reached a particular concentration (10 mg-S/L). The two strategies of sulfide control were carried out by intermittent nitrate dosing. The carbon, nitrogen, and sulfur cycles in the process were investigated during the experiments.

The optimal dosing control condition was determined by short-term batch tests. The consistent sediment environment and the same initial condition were kept for serial test

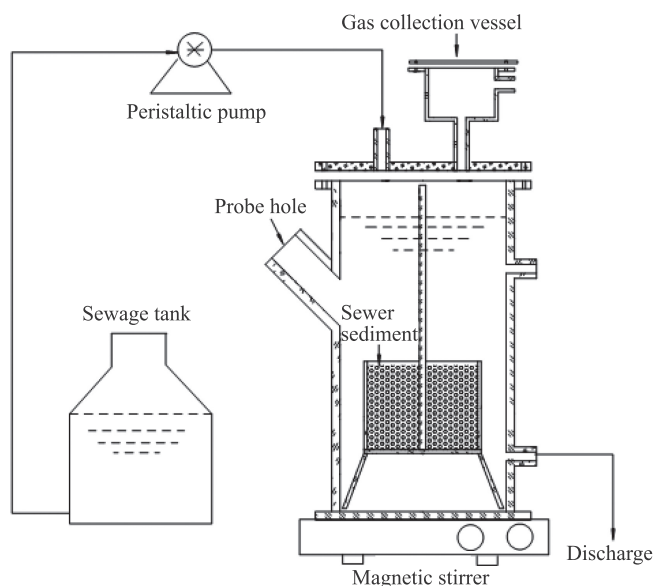


Fig. 1 – Schematic of the lab-scale sewer reactor.

experiments. The carbon, nitrogen, and sulfur cycle rates were also evaluated during the experiments. The initial concentrations of sulfide and nitrate in different reactors (listed as Test 1, Test 2 and Test 3) were 6.5, 13.0, 17.5 mg-S/L and 11.0, 9.0, 6.2 mg-N/L, respectively. The initial concentrations of COD and VFA in Test 1, Test 2 and Test 3 were 185.8, 167.4, 184.0 mg-COD/L and 7.4, 3.9, 4.6 mg-COD/L, respectively. The optimal control condition was determined by the influence of different ratios of nitrate per sulfide (N/S) (1.70, 0.70 and 0.35) on sulfide removal rate, nitrate denitrification rate, and rate of carbon source loss, respectively.

### 1.3. Chemical analysis method

For the analysis of dissolved sulfur species (sulfite, thiosulfate, sulfate), the wastewater was pre-treated with zinc acetate to precipitate sulfide. The wastewater was then fixed using 4% formaldehyde to protect the sulfite. The samples were then analyzed using an ion chromatograph (IC) with ultraviolet (UV) (Dionex ICS-1000) (SEPA, 2002). Before the analysis of sulfide, the samples were immediately conserved by adding zinc acetate and adjusting the pH value using sodium hydroxide to keep the pH same as the standard samples. Sulfide was detected by the methylene blue method (Ju and Tao, 2007) using a spectrophotometer (HACH, USA, DR5000). For the analysis of the nitrogen species (nitrate, nitrite), a UV spectrophotometer was used. VFA content was measured by Agilent gas chromatography with an analyte protectants free fatty acids column, following the method of Gutierrez et al. (2008). The VFA species included acetate, propionate, butyrate, isobutyrate, valerate, and isovalerate. The measured values were converted to COD by the theoretical COD values of these chemicals. The soluble and total COD contents were determined using a COD digestion apparatus according to standard methods (APHA, 1998). ORP was measured with an ORP sensor (Mettler Toledo, Switzerland, LE501) connected to an ORP transmitter. The pH was determined by a pH meter (Mettler Toledo, Switzerland, FE20).

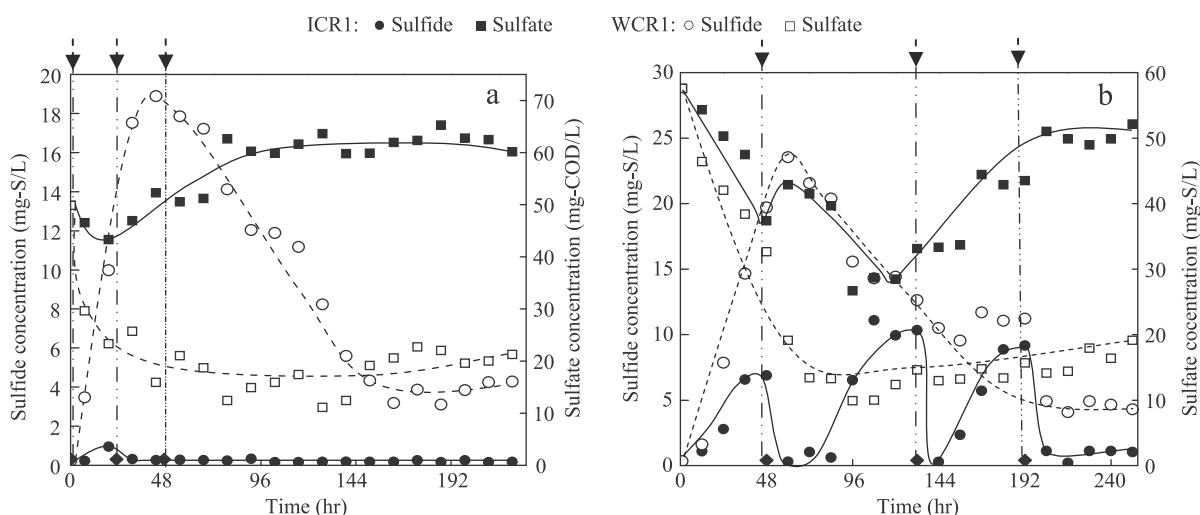
## 2. Results and discussion

### 2.1. Effect of sulfide control by different nitrate dosing strategy

The sulfide accumulation (reaching approximately 20 mg-S/L) and the sulfate reduction were observed in the reaction process of the reactors WCR1 and WCR2 during the entire experiment. In the first SFE experiment, the sulfide concentration was kept below 0.3 mg-S/L in ICR1 reactor (Fig. 2a). The sulfate concentration was kept at a particular level, which means that the specific reduction of sulfate in the sediment was completely inhibited by the nitrate that was exposed for prolonged periods of time in bulk water. By contrast, in the second SIE experiment, the relatively slow accumulation rate of sulfide immediately decreased to a negligible level after every nitrate dosing event in the ICR2 reactor (Fig. 2b). The accumulation rate of sulfide eventually decreased to a very low level after third dosing event. However, the sulfate concentrations decreased as the sulfide accumulation process was on-going. This result indicates that the reduction of sulfate in the sediment was not completely inhibited after nitrate dosing. Moreover, the sulfate concentration returned to its initial value at the end stage of the experiment. The result indicates that no sulfide emission happened from the bulk water. The SIE control strategy can achieve the same effective control of the SFE control strategy by intermittently hindering the sulfate reduction ability. However, the SIE control strategy consumed relatively fewer nitrates.

### 2.2. Influence of different nitrate dosing strategy on loss of carbon source

The excess carbon source is necessary for denitrification process in wastewater treatment plant. Minimizing the loss of carbon source should be considered during the operation of drainage systems. In the WCR reactors, sulfide was accumulated when sulfate was strongly reduced in the first period. After that, the



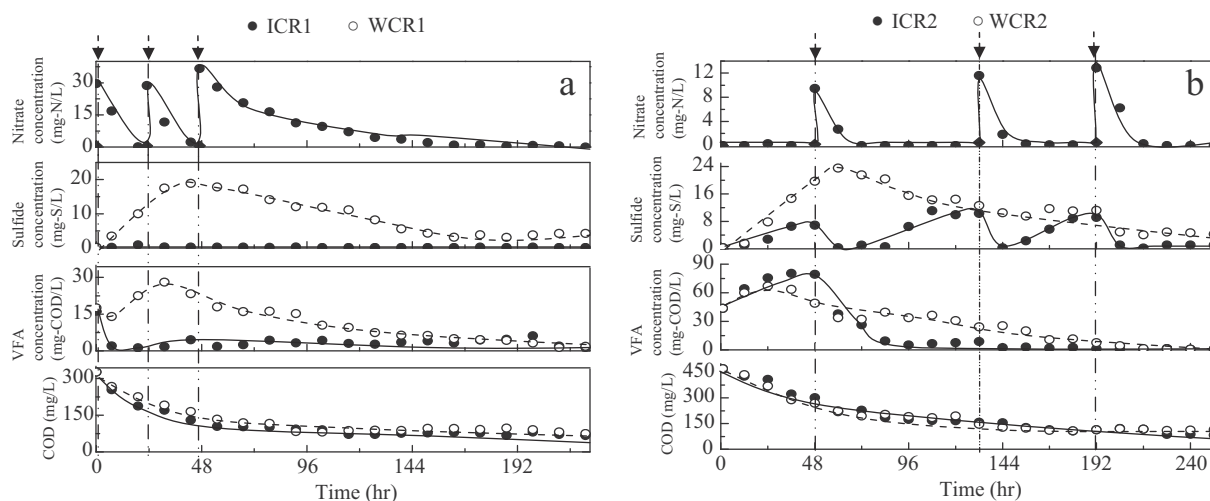
**Fig. 2 – Sulfur concentration change in implement control reactor (ICR)1 and without control reactor (WCR)1 (a) and implement control reactor (ICR)2 and without control reactor (WCR)2 (b). The arrows indicate the nitrate dosing events.**

concentration of sulfide began to decline because of sulfide emission from liquid phase while the reduction of sulfate weakened (Fig. 2). The profile of VFA was similar to that of the sulfide, but the turning point of VFA where it began to decline occurred earlier compared to that of sulfide (Fig. 3). The reduction of sulfate was weakened when the VFA was maintained at a relatively high concentration (23.3 and 33.8 mg-COD/L). The results indicate that VFA accumulation cannot significantly enhance the reduction of sulfate, although a high amount of VFA is beneficial for sulfate reduction as it functions as an essential electron donor.

The VFA concentration quickly declined after nitrate was added into the bulk, and it was kept at low concentrations (6.0 and 8.7 mg-COD/L) in both nitrate dosing control experiments (Fig. 3). The phenomenon further revealed that nitrate is more competitive than sulfate (Hvitved et al., 2000). The decline of

sulfide concentrations to lower levels occurred prior to the decline of VFA in the SIE control experiment (Fig. 3b). This result indicates that sulfide was preferentially used in nitrate reduction. Furthermore, the sulfide continued to accumulate (up to 10 mg-S/L) when the VFA concentration was very low after the second dosing nitrate event in the SIE experiment. The result further revealed that the accumulation amount of VFA was not the limiting factor for the reduction of sulfate. Previous study by Jiang et al. (2009) also showed that VFA (20–100 mg-COD/L) had no effects on elemental sulfur and on the sulfate reduction process.

The sediments in sewers are very complex. Multispecies biofilms and microorganisms, multiple electron donors (sulfide and COD), and electron acceptors (nitrate and sulfate) are present in their physico-chemical microenvironments. Nitrate denitrification, sulfate reduction, and sulfide



**Fig. 3 – Changes in sulfur, nitrogen, and organic compound in implement control reactor (ICR)1 and without control reactor (WCR)1 (a) and implement control reactor (ICR)2 and without control reactor (WCR)2 (b). The arrows indicate the nitrate dosing events.**

**Table 1 – Reaction rate in long-term performance experiments.**

Control strategy	Dosing events	implement control reactor (ICR)			without control reactor (WCR)	
		$r_{\text{NO}_3}$ (mg-N/(L·hr))	$r_{\text{COD}}$ (mg-COD/(L·hr))	$r_{\text{VFA}}$ (mg-COD/(L·hr))	$r_{\text{COD}}$ (mg-COD/(L·hr))	$r_{\text{VFA}}$ (mg-COD/(L·hr))
SFE	First	1.53	7.10	0.65	5.15	0.31 <sup>a</sup>
	Second	1.14	3.40	0.23	2.24	0.39
	Third	0.28	0.20	0.00050	0.19	0.080
SIE	–	–	3.96	1.03	5.21	0.56 <sup>a</sup>
	First	0.55	1.66	0.91	0.92	0.14
	Second	0.71	1.18	0.10	0.77	0.33
	Third	0.62	0.69	0.0030	0.060	0.11

<sup>a</sup> Rate is positive.

oxidation processes can simultaneously occur. The consumption of the carbon source in the WCR reactor was regarded as the basic carbon consumption rate (for sulfate reduction and normal metabolism), and the carbon source consumption in the ICR reactor was reinforced by nitrate denitrification. When the differences in COD consumption rate between WCR and ICR reactors shown in Table 1 are compared with each other, the difference in the SFE control strategy experiment is significantly larger than in the SIE control strategy experiment. The result indicates that the SFE control strategy consumed more carbon sources because of nitrate denitrification. However, most of the nitrate was used for the oxidation of sulfide in the SIE control strategy. The SIE control strategy can achieve the same sulfide control effect with minimum carbon source loss.

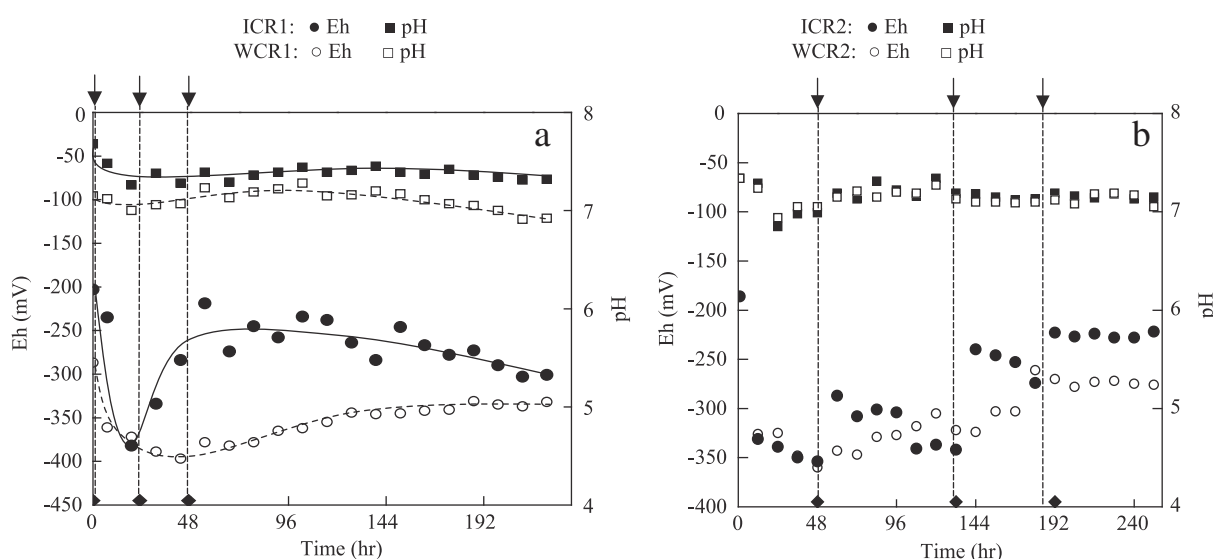
### 2.3. Parameter response of sulfide elimination

According to typical sulfur cycle theory, the anaerobic environment parameters (such as pH and ORP) are the critical factors for the occurrence of sulfur cycle (Nielsen et al., 1992; Hvitved et al., 2000). ORP less than –320 is a conducive condition for sulfate

reduction (Wieringa, 1940). The changes in the profile of ORP correspond well with the sulfide accumulation curve in the WCR reactors in different control strategy experiments (Figs. 2 and 4). The ORP gradually decreased with the accumulation of sulfide because sulfide has a strong reduction ability (Poduska and Anderson, 1981). Moreover, the ORP gradually increased after nitrate was added in the ICR reactors because of the presence of nitrate in the water. The increase in ORP continued until the sulfide began to accumulate (Fig. 4). The result indicates that the change in ORP is a good indicator of the accumulation status of sulfide during the control process that reflected well the control effect of the SIE control strategy. However, the pH did not significantly vary during the reaction process of the different sulfide control strategies. The pH only decreased slightly (7.5–7.0) in the initial phase of the SIE control experiment because of the accumulation of VFA.

### 2.4. Optimal operation control condition of sulfide intermittent elimination

The accumulated sulfide concentration and the nitrate dosing amount of every dosing event must be considered in the SIE



**Fig. 4 – Parameter measurements as indicator of sulfide control in implement control reactor (ICR)1 and without control reactor (WCR)1 (a) and implement control reactor (ICR)2 and without control reactor (WCR)2 (b). The arrows indicate the nitrate dosing events.**



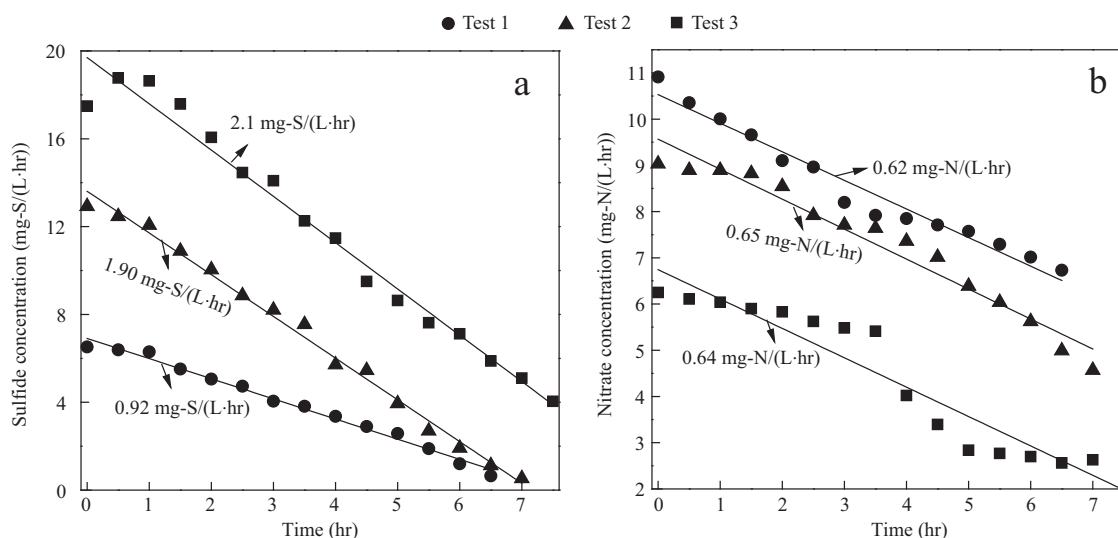


Fig. 5 – Sulfide removal rates (a) and nitrate reduction rates (b) in Test 1, Test 2, and Test 3.

control strategy. The optimal dosing condition was determined by batch tests. The result is shown in Fig. 5, and the typical reaction rates are summarized in Table 2. The result indicates that the sulfide removal rate significantly increased with increasing accumulated sulfide concentration and with decreasing N/S ratio (from 1.7 to 0.7) if nitrate was kept in sufficient excess. However, the nitrate denitrification rate was constant (data of Test 1 and Test 2 shown in Fig. 5) in the experiment. This result indicates that the rate of sulfide oxidation was dependent on sulfide concentration and N/S ratio when nitrate was in excess. This result corresponds with those of a previous study (Yang et al., 2005). However, sulfide removal and nitrate denitrification rates remained high when the N/S ratio was further decreased (from 0.7 to 0.35) until sulfide was in excess. However, the sulfate production rate significantly decreased (Table 2) because of the incomplete oxidation of sulfide to sulfate. Thus, other sulfur intermediates were formed as the important end product which was discussed and hypothesized in previous studies (Jiang et al., 2009; Nielsen et al., 2005). The result indicates that a high sulfide removal rate can be maintained with sulfur intermediates as the final oxidation product at a low N/S ratio in the SIE control strategy.

The influence of N/S ratio on carbon source degradation is shown in Table 2. The VFA degradation rate significantly decreased with decreasing N/S. This result indicates that nitrate denitrification weakened with decreasing N/S ratio because the dosing nitrate amount was decreased or the sulfide amount was increased. This means the low N/S ratio can promote low carbon source loss. Therefore, a slight excess of nitrate dosing amount is necessary in the SIE control strategy to ensure that the sulfide is

completely removed. However, minimizing the dosage of nitrate can reduce the N/S ratio, which is beneficial for promoting the sulfide removal rate and reducing the carbon source loss. An N/S ratio range of 0.5 to 0.6 is recommended as the optimal control condition of the SIE control strategy in this study. The result is slightly higher than the theoretical N/S ratio for oxidizing sulfide to sulfur, which is estimated to be 0.18–0.44 (Zhang et al., 2008; Yang et al., 2005).

### 3. Conclusion

A cost-effective control strategy and the optimal condition of intermittent nitrate dosing for sulfide elimination in sewer sediments were investigated. The newly proposed SIE strategy by intermittent nitrate dosing based on the change in the dissolved sulfide content in sewers was preferred. The optimal control condition includes a minimum N/S ratio to ensure that nitrate is in excess (N/S of 0.5–0.6). The maximum sulfide removal rate and the minimum carbon source loss can be simultaneously achieved in the optimal control condition. The ORP is a sensitive indicator of the control effect for the implementation of the SIE strategy.

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Table 2 – Reaction rates under different nitrate/sulfide (N/S) conditions.

Reactor	N/S	$r_{\text{sulfide}}$ (mg-S/(L·hr))	$r_{\text{nitrate}}$ (mg-N/(L·hr))	$r_{\text{sulfate}}$ (mg-S/(L·hr))	$r_{\text{COD}}$ (mg-COD/(L·hr))	$r_{\text{VFA}}$ (mg-COD/(L·hr))
Test 1	1.70	0.92	0.62	1.77	11.44	0.85
Test 2	0.70	1.90	0.65	2.38	10.88	0.58
Test 3	0.35	2.10	0.64	1.02	11.84	0.33

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