


Enhanced sulfide removal by gas stripping in a novel reactor for anaerobic wastewater treatment

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ABSTRACT

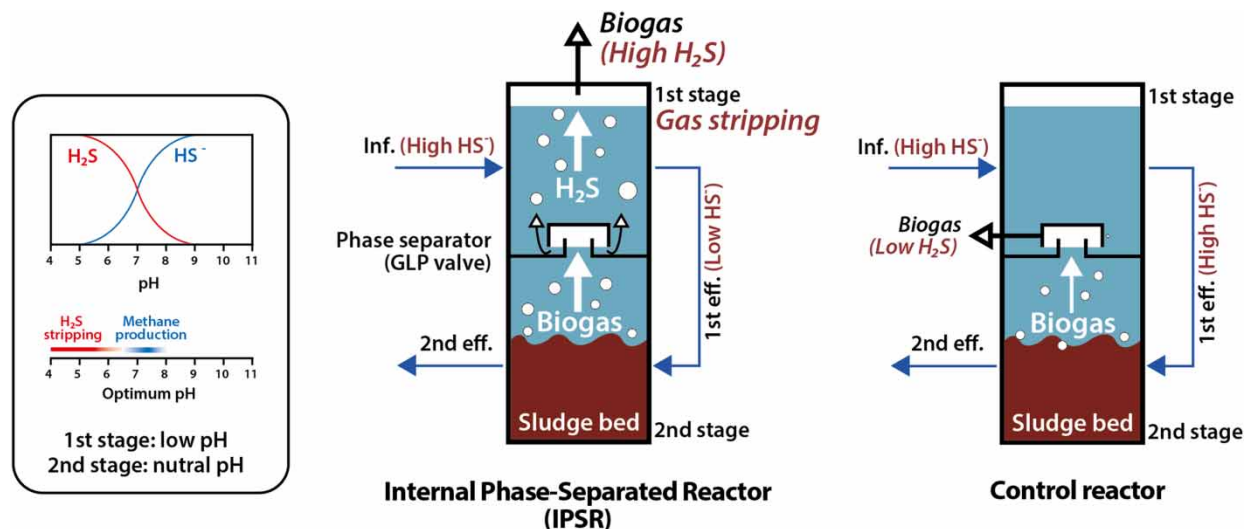
Removal of sulfide by gas stripping using biogas produced in an internal phase-separated reactor (IPSR) was evaluated during anaerobic treatment. The IPSR consisted of upper and lower segments with a gas–liquid partitioning (GLP) valve between the sections. Wastewater was fed to the upper segment in the first stage and then to the lower segment in the second stage. The GLP valve separated the liquid phase from the gaseous phase and supplied biogas from the lower segment to the upper segment. The IPSR and a control reactor were fed with synthetic wastewater and operated in parallel under an organic loading rate of 12 kg COD/(m³ day) at 35 °C. The sulfide concentration increased to 400–600 mg S/L, which is above the previously reported 50% inhibition level for methanogenic activity. The IPSR showed higher H₂S removal performance than the control reactor and removed approximately twice the H₂S as the control reactor at 400 mg S/L, indicating that it can be used for the stable treatment of wastewater containing high concentrations of sulfide.

Key words: anaerobic treatment, gas stripping, inhibition, sulfide removal, two-phase process

HIGHLIGHTS

- Sulfide removal was evaluated in an internal phase-separated reactor (IPSR).
- The IPSR consisted of an upper segment (first stage) and a lower segment (second stage).
- Sulfide in the upper segment was removed by biogas supplied from the lower segment.
- Sulfide removal was higher in the IPSR than in the control reactor.
- The IPSR had higher organic removal than the control reactor.

GRAPHICAL ABSTRACT



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1. INTRODUCTION

Much attention has been paid to anaerobic wastewater treatment processes because they enable the recovery of energy from waste organics via methane gas. To enable anaerobic treatment, inhibition caused by ammonia, sulfide, light metals, heavy metals, and organic materials needs to be addressed. These inhibitors are often found in wastewater or produced during anaerobic digestion; therefore, the incorporation of methods to eliminate inhibitors can result in higher treatment efficiency and process stability (Chen *et al.* 2008; Yuan & Zhu 2016).

Sulfide in the liquid phase is toxic to methanogens and inhibits anaerobic processes (Karhadkar *et al.* 1990; Chen *et al.* 2008). Sulfide can be a component of wastewater or produced by sulfate-reducing bacteria. Sulfide and sulfate are often found in industrial wastewaters produced by the tannery, pulp, and paper industries (Wiemann *et al.* 1998; Hao *et al.* 2014). The 50% inhibition level for methanogenic activity has been reported to be 90–250 mg S/L under different pH conditions (Koster *et al.* 1986). Other studies have reported even higher inhibition levels. For instance, 50% inhibition of methanogenic activity was also observed at a sulfide concentration of 500 mg S/L and pH of 7.0–7.2 (Karhadkar *et al.* 1990) and at 650 mg S/L and pH 7.5 for acetate-utilizing methanogens (Onodera *et al.* 2011). Notably, free hydrogen sulfide (H_2S) is more toxic to methanogens than hydrosulfide ion (HS^-) (Koster *et al.* 1986).

A strategy for minimization of free H_2S is important to successful anaerobic treatment of wastewater (Omil *et al.* 1995). To reduce free H_2S , pH control or additional processes for sulfide removal are required. Sulfide removal processes include chemical reactions (oxidation and precipitation), physico-chemical techniques (gas stripping), biological conversions (elemental sulfur production), and combined techniques (Chen *et al.* 2008; Li *et al.* 2015). In two-stage processes, sulfide removal can be applied via acidification in the first stage and methane production in the second stage (Stefanie *et al.* 1994). Acidification lowers the pH and increases the $\text{H}_2\text{S}/\text{HS}^-$ ratio, allowing the gas-stripping process to reduce sulfide more effectively in the liquid phase (Omil *et al.* 1995; Wei *et al.* 2007). Although gas-stripping processes are effective, they require mechanical devices and energy consumption (Hao *et al.* 2014).

In this study, we investigated the use of biogas produced during anaerobic digestion for gas stripping of H_2S . To accomplish this, we developed a novel concept in an anaerobic reactor consisting of a gas-stripping stage, methane fermentation stage, and a phase separator (Onodera *et al.* 2022). In this system, the gas stripping and methane fermentation stages were arranged vertically and connected by a phase separator that consisted of a newly designed gas–liquid partitioning (GLP) valve. The critical function of the GLP valve is to block liquid flows between the stages while allowing biogas generated by methane fermentation in the lower segment to enter the upper segment for gas stripping. Unlike conventional gas-stripping processes (Stefanie *et al.* 1994), the GLP valve allows gas stripping without the need for mechanical devices (e.g., blowers and diffuser pipes) and electric power. The developed system was named an internal phase-separated reactor (IPSR). The first study of an IPSR fed with high-sulfate wastewater revealed higher H_2S removal via biogas and lower sulfide concentrations in the effluent under a high organic loading rate (OLR) of 10 kg COD/(m^3 day) (Onodera *et al.* 2022). Thus, the IPSR may be a powerful treatment technology that can be applied to many types of wastewaters. However, little is known about the process performance with respect to various types of wastewater, such as sulfide wastewater. It is also important to continue to demonstrate the technical superiority of IPSR under different conditions.

Here, we applied the IPSR for high-sulfide wastewater and evaluated its feasibility for H_2S removal using biogas produced in the IPSR. Both reactors were operated in parallel and fed with synthetic wastewater containing sulfide at 400–600 mg S/L. The IPSR was operated without recirculation and directly fed with sulfide at 400–600 mg S/L so that the effects of the GLP valve on H_2S removal could be evaluated directly.

2. METHODS

2.1. Internal phase-separated reactor

The IPSR and control reactor are depicted in Figure 1. Laboratory-scale cylindrical reactors were used. The IPSR consisted of an upper segment as the first stage, a phase separator, and a lower segment as the second stage. The heights of the first stage, phase separator, and second stage were 1.10, 0.15, and 1.05 m, respectively. The diameter of the segments was 0.079 m. A GLP valve was used as the phase separator. The inner and outer diameters of the GLP valve were 0.016 and 0.041 m, respectively. The working volumes in the first and second stages were 5.35 and 5.15 L, respectively. The temperature of the reactor was controlled at 35 °C using a water jacket. The GLP valve transferred biogas produced in the second stage (lower segment)

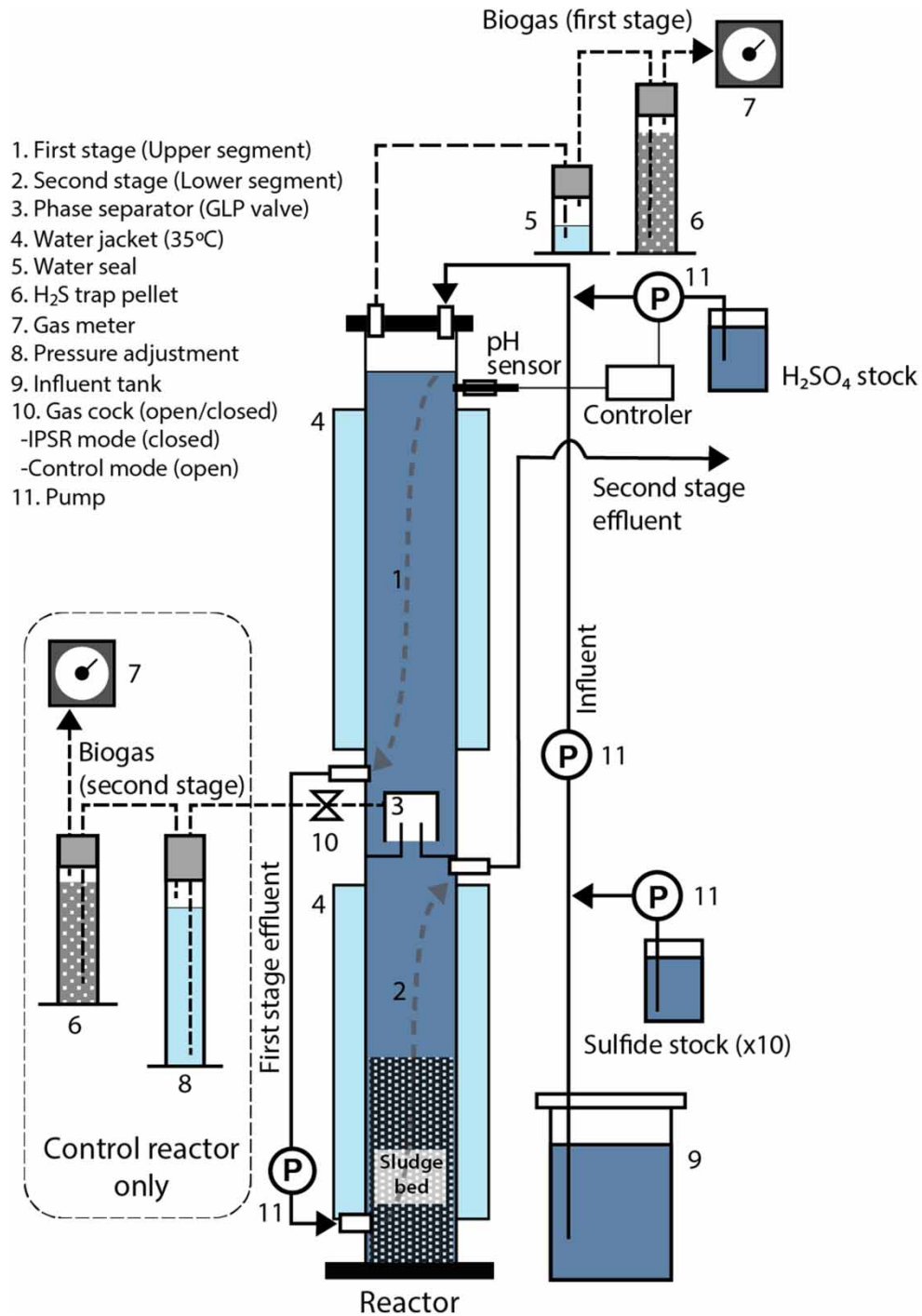


Figure 1 | Schematic diagram of the laboratory-scale IPSR with a GLP valve and the control reactor.

to the first stage (upper segment) of the reactor while blocking the flow of liquid between segments. The function of the GLP valve can be changed by opening and closing the gas cock.

In the first stage, which did not contain sludge, sulfide was removed by gas stripping using biogas. The second stage, which was used for methane fermentation, was filled with mesophilic granular sludge obtained from an upflow anaerobic sludge blanket (UASB) reactor fed with synthetic wastewater. The UASB was operated at an OLR of 20 kg COD/(m³ day) and the sludge was taken on day 677. Details regarding the operating conditions and reactor performance of the UASB are

presented elsewhere (Onodera *et al.* 2022). Operation started within 30 min of the sludge being transferred to the reactors. The total solid (TS) concentration of the sludge was 70.9 g/L, while the volatile solid (VS) concentration was 63.5 g/L (VS/TS = 0.90) and the sludge volume index was 2 mL/g TS. The total volume of the sludge was 4.0 L (284 g TS and 254 g VS for each reactor). After the reactor experiment, the sludge in the IPSR and the VS contained 163 g VS (189 g TS) and 149 g VS (199 g TS), respectively.

2.2. Operating conditions

The reactor operating conditions are shown in Table 1. During the first period, the reactors were operated without feeding high concentrations of sulfide (Na_2S). The comparative experiment was conducted after the second period. The configurations of the IPSR and the control reactor were the same except for the mode of the phase separator (Figure 1). In the IPSR, biogas produced in the second stage moved to the first stage, after which it was released. In the control reactor, biogas was released from the middle portion of the reactor via the GLP valve (Figure 1).

2.3. Synthetic wastewater

The reactor was fed with synthetic wastewater under the operating conditions described in Table 1. The wastewater concentrations shown in the table were determined after mixing with sulfide solution ($10\times$) consisting of $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$. The organic substances in the synthetic wastewater consisted of 2,400 mg/L sucrose, 1,800 mg/L CH_3COONa , 900 mg/L propanoic acid, and 600 mg/L peptone. Additionally, the wastewater contained 320 mg/L Na_2SO_4 , 4,000 mg/L NaHCO_3 , 150 mg/L KH_2PO_4 , 150 mg/L $\text{CaCl}_2\cdot 2\text{H}_2\text{O}$, 400 mg/L $\text{MgCl}_2\cdot 6\text{H}_2\text{O}$, 300 mg/L KCl, 110 mg/L NH_4Cl , and a trace element solution. The trace elements solution consisted of 3.93 mg/L $\text{FeCl}_2\cdot 4\text{H}_2\text{O}$, 0.17 mg/L $\text{CoCl}_2\cdot 6\text{H}_2\text{O}$, 0.07 mg/L ZnCl_2 , 0.06 mg/L H_3BO_3 , 0.5 mg/L $\text{MnCl}_2\cdot 4\text{H}_2\text{O}$, 0.04 mg/L $\text{NiCl}_2\cdot 6\text{H}_2\text{O}$, 0.03 mg/L $\text{CuCl}_2\cdot 2\text{H}_2\text{O}$, and 0.03 mg/L $\text{NaMoO}_4\cdot 2\text{H}_2\text{O}$. This wastewater composition was based on an established method (Sekiguchi *et al.* 1998). The pH in the influent was adjusted to approximately 6–7 using H_2SO_4 solution, after which an anti-foaming reagent (Antifoam SI, FUJIFILM Wako Pure Chemical Corporation, Osaka, Japan) was added. The synthetic wastewater was prepared once a week and kept at room temperature (approximately 26–27 °C).

2.4. Analysis

Water samples were taken from the influent, first segment effluent, and second segment effluent. The pH and oxidation–reduction potential (ORP) were determined immediately after sampling using a pH meter (9625-10D, D-74, LAQUA, Kyoto, Japan) and an ORP meter (9300-10D, D-74, LAQUA), respectively. For dichromate COD (COD_{Cr}) analysis, the sample was amended with H_2SO_4 , then purged with N_2 to remove the sulfide. After pre-treatment of samples, the COD was measured using TNT822 and TNT823 test kits (Hach, Loveland, CO, USA). The SO_4 concentration was determined using SulfaVer4 (2106769, Hach). The COD_{Cr} and SO_4 were measured using a Hach water quality analyzer (DR1900, Hach). The produced biogas was collected in a gas bag after H_2S trap and the volume was measured with a wet-test gas meter (WS – 1A, Shinagawa, Tokyo, Japan). The biogas composition was determined using a gas analyzer (Biogas 5000, Geotech, Geotechnical Instruments Ltd, Leamington Spa, UK). The H_2S in the gas before H_2S trap was measured using a gas detector tube system (4HP and 4HH, GV-100S, GASTEC, Kanagawa, Japan). The biogas production was determined after

Table 1 | Operating conditions of the IPSR and control reactor

Parameter	Unit	1st period	2nd period	3rd period	4th period
Operating period	days	6	7	9	4
Temperature	°C	35	35	35	35
Flow rate	L/day	10.30	10.30	10.30	10.30
HRT (second stage)	hr.	12	12	12	12
Influent COD concentration	mg COD/L	6,000	6,000	6,000	6,000
COD loading (second stage)	kg COD/(m ³ day)	12.0	12.0	12.0	12.0
Influent sulfate concentration	mg S/L	72	72	72	72
Influent sulfide concentration	mg S/L	0	400	500	600

H₂S trap. The volume of biogas and methane produced was calculated using standard conditions. Other analyses were conducted on the basis of standard methods (APHA 2005).

3. RESULTS AND DISCUSSION

3.1. Comparative performance of IPSR and control reactor

The performance of the IPSR and the control reactor in terms of pH, COD, biogas production, and CH₄ production is shown in Figure 2. The first period is preliminary operation before the performance comparison. As expected, the IPSR and control reactor showed stable process performance at an OLR of 12 kg COD/(m³ day) during the first period. The COD in the second effluent was low, while high biogas and CH₄ production was observed in the first period. The pH of the second stage effluent from both reactors was neutral, indicating that these conditions were appropriate for use in comparative experiments of sulfide removal by both reactors. The first stage effluent was maintained at low pH conditions, which are suitable for H₂S removal by gas stripping (Omil *et al.* 1995; Wei *et al.* 2007).

In the second period, the influent sulfide concentration was 400 mg S/L. The results showed that the performance of the IPSR was better than that of the control reactor. The COD of the second effluent was approximately 500 mg/L for the IPSR and over 1,000 mg/L for the control reactor. The biogas and CH₄ production were also lower in the control reactor than the IPSR. Moreover, the control reactor showed an increase in COD in the second effluent and considerably decreased biogas and CH₄ production in the third period. Thus, COD removal was related to biogas and CH₄ production. The pH in the second effluent was also lower, while the ORP was increased in the third period. These findings indicate that the process had collapsed in the control reactor because of sulfide inhibition. The IPSR also had a slightly higher COD concentration and lower biogas and CH₄ production when the performance in the second period was compared, but maintained superior performance compared with the control reactor. The pH was neutral and the ORP was less than -400 mV in the second effluent of the IPSR, indicating that the IPSR could tolerate high influent sulfide levels in the second (400 mg S/L) and third period (500 mg S/L).

3.2. H₂S removal by biogas

The H₂S concentration in the biogas is shown in Figure 3(a). Biogas was produced in the second stage in both reactors; however, this gas was fed into the first stage in the IPSR while it was released from the second stage in the control reactor. In the first period, when no sulfide was added to the influent wastewater, the H₂S concentration in the biogas was less than 1.2%. During this period, H₂S in the biogas was produced by sulfate reduction using influent sulfate in the synthetic wastewater. In the second period, the H₂S concentration in the biogas from the IPSR was higher than that from the control reactor. In the third period, the biogas H₂S concentration increased in both reactors. The H₂S concentrations in the control reactor were found to be similar to those in the IPSR during the third period. This was attributed to a decrease in the amount of biogas produced in the control reactor as well as higher sulfide concentrations in the second stage. The H₂S removed based on wastewater volume is shown in Figure 3(b). The results clearly indicate that the IPSR had superior H₂S removal than the control reactor. In addition, the IPSR showed higher H₂S removal when the influent sulfide was higher (third period).

3.3. Relationship between biogas production rate and H₂S removal

The relationship between biogas production rate and H₂S removal based on the wastewater volume is shown in Figure 4. The H₂S removed was calculated based on the biogas volume and the H₂S composition. The IPSR had higher H₂S removal than the control reactor, even when the biogas production rate was the same. This indicates that the biogas produced in the second stage of the IPSR was efficiently used for H₂S stripping and had high H₂S removal performance. In the control reactor, the H₂S removal was correlated with the biogas production; therefore, decreased biogas production results in decreased H₂S removal. The lower H₂S removal rate is thought to have occurred via a negative chain reaction in which the sulfide inhibition was intensified, causing decreased biogas production. Because H₂S removal occurred in the second stage in both the IPSR and the control reactor, the higher H₂S removal in the IPSR can be attributed to additional H₂S removal in the first stage. Specifically, the IPSR can greatly improve H₂S removal by introducing biogas from the second stage to the first stage while maintaining the optimum pH conditions for gas stripping and methane fermentation in the separate stages.

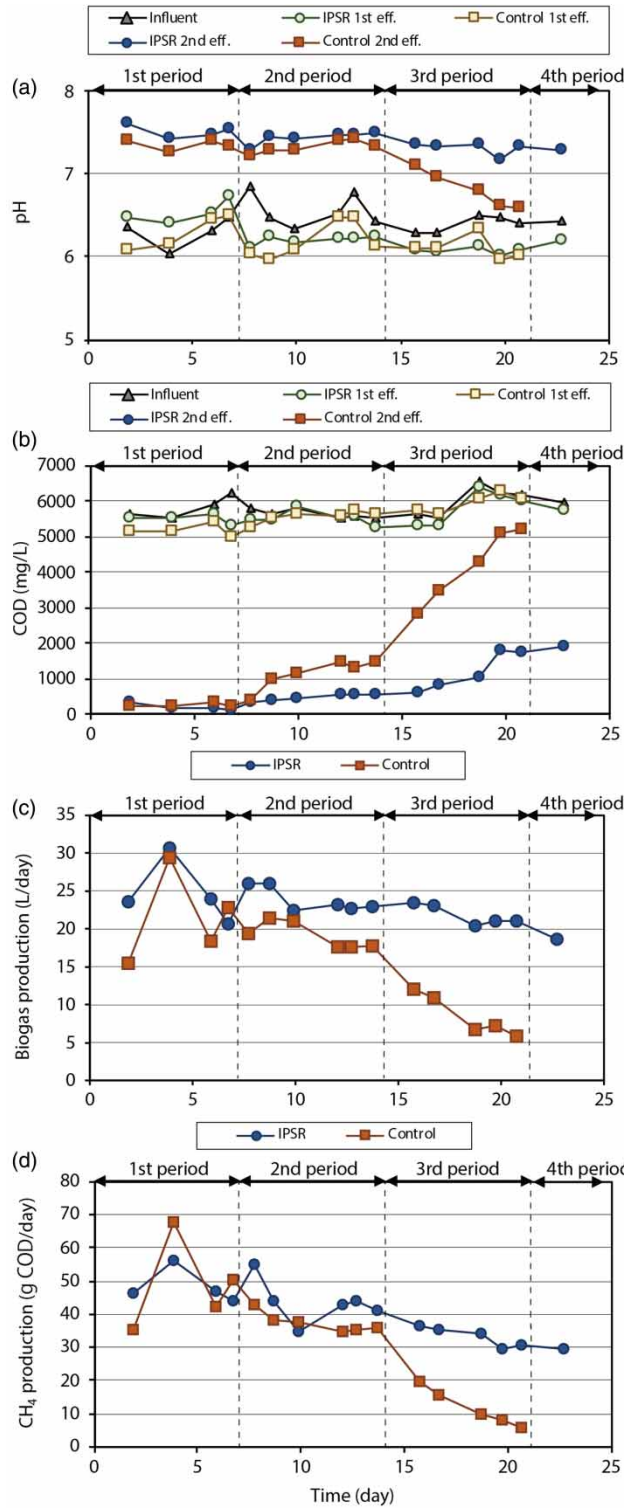


Figure 2 | Comparison of the IPSR and the control reactor: (a) pH, (b) COD, (c) biogas production and (d) methane production.

3.4. H₂S removal in the IPSR and control reactors

The average sulfur concentrations in the IPSR and control reactors in the second and third period are shown in Figure 5. The influent sulfur was the sum of sulfate and sulfide. The sulfate was completely reduced in the reactor and could produce sulfide via sulfate reduction. Here, the effluent sulfide from the IPSR or control reactor was calculated as influent sulfur (sulfide and

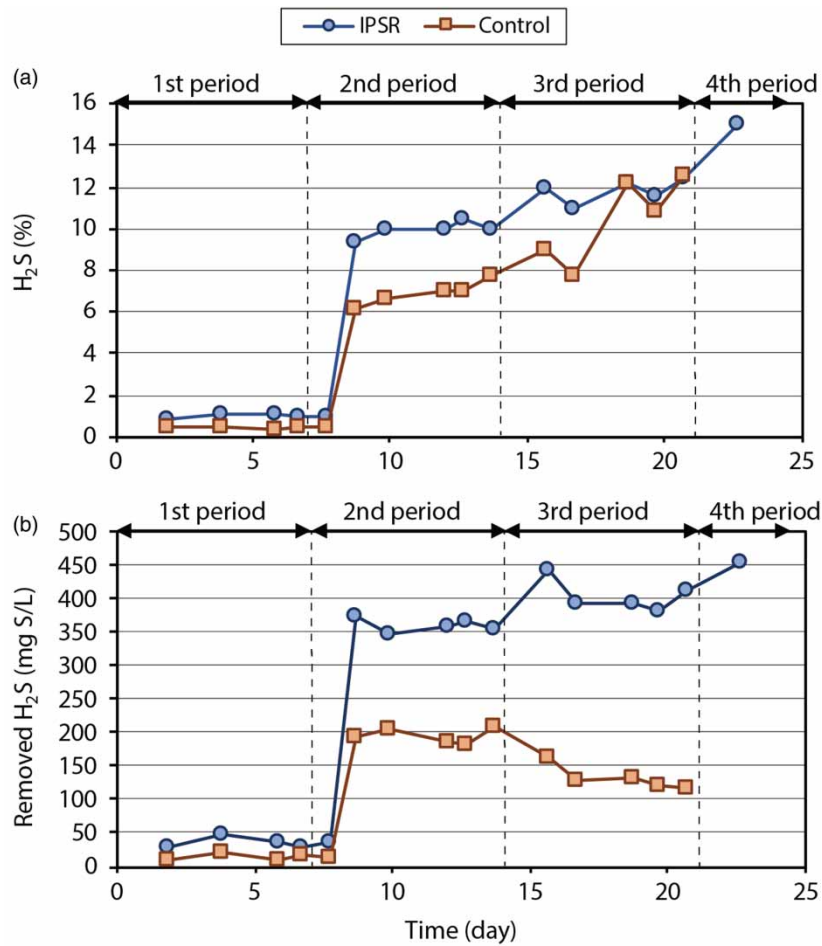


Figure 3 | Comparison of the IPSR and the control reactor: (a) H_2S concentration in biogas and (b) H_2S removed based on the wastewater volume calculated from biogas production and the H_2S concentration.

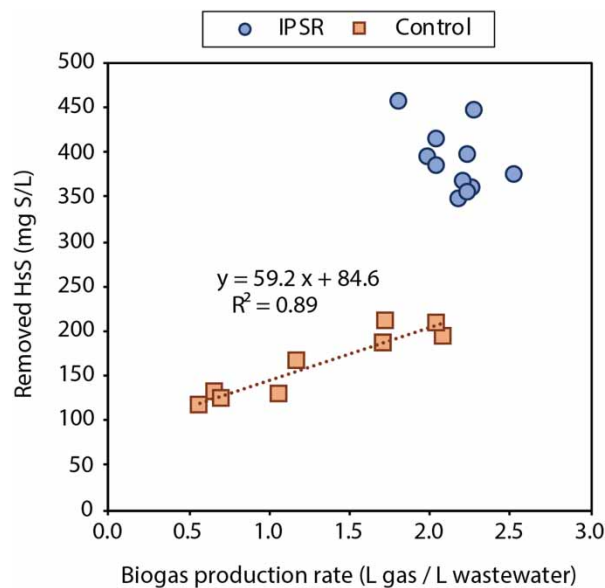


Figure 4 | Relationship between biogas production rate and H_2S removal in the IPSR and the control reactor.

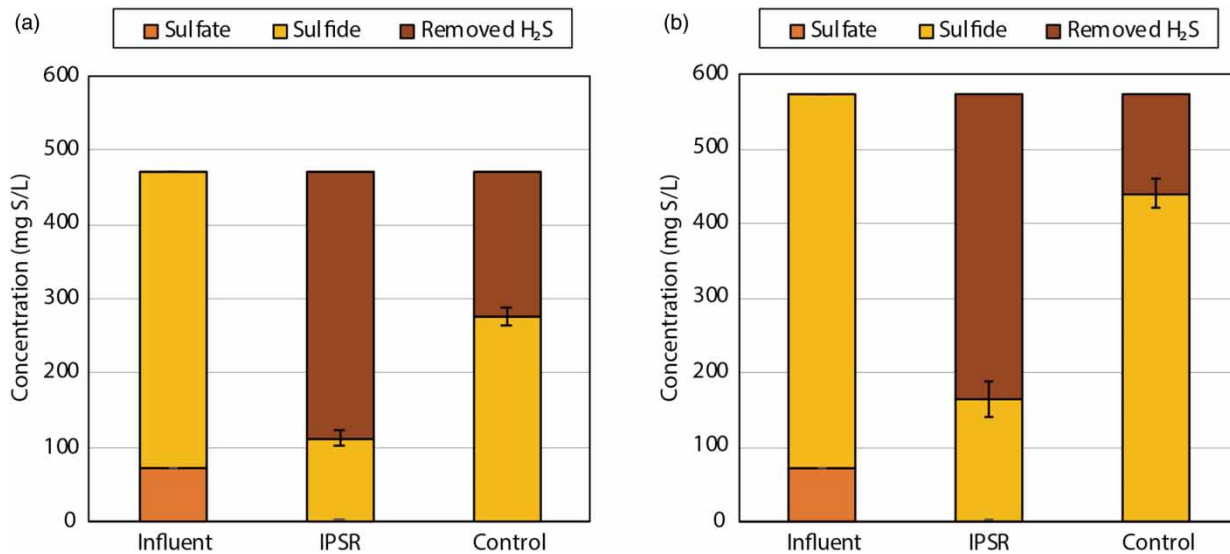


Figure 5 | Sulfur concentration in the IPSR and the control reactor during the second (a) and third periods (b).

sulfate) minus H₂S removed in the biogas. These findings indicate that the IPSR produced effluent with lower sulfide concentrations than the control reactor. Indeed, as shown in Figure 5, the IPSR removed approximately twice the H₂S as the control reactor in the second period. Additionally, the IPSR showed nearly three times the H₂S removal rate as the control reactor in the third phase, although it is important to note that they were not completely comparable because of failure of the control reactor during this phase. The unique reactor structure of the IPSR contributed to the dramatic increase in H₂S removal rates. High sulfide concentrations suppress microbial activity, especially at lower pH (Koster *et al.* 1986); therefore, process performance relies on sulfide removal in anaerobic reactors. Because of its high sulfide removal, the IPSR tolerated high sulfide concentrations in the influent wastewater.

3.5. Applicability of IPSR

The IPSR removed hydrogen sulfide from the wastewater by gas stripping, thereby reducing the low concentration of sulfide in the wastewater. As a result, sulfide inhibition in the methanogenic phase is expected to be alleviated. Instead of reduced sulfide concentrations in the wastewater, the biogas produced contained high concentrations of hydrogen sulfide. To enable biogas utilization for electricity generation or upgrading to biomethane, H₂S must be removed from the biogas (Choudhury *et al.* 2019). Various treatment methods for desulfurization of biogas have been proposed in previous studies (Lupitskyy *et al.* 2018). However, an appropriate technology for H₂S removal from biogas produced in an IPSR needs to be selected as a post-treatment of biogas during its application.

Several methods for removal of sulfide from wastewater have been proposed, including physical methods, chemical reactions, and biological transformations (Chen *et al.* 2008; Robles *et al.* 2020). However, many of these techniques incur additional costs because they require chemicals (e.g., precipitation) or mechanical operations (e.g., gas stripping using air or N₂). Therefore, the primary advantage of IPSR is that it is a simple and low-cost process. This is because the biogas produced by the IPSR can be used for gas stripping and introduced naturally into the reactor. Although sulfide removal in the IPSR was sufficient in this study, further sulfide removal may be difficult to control because it depends primarily on the amount of biogas produced. To enable application to wastewater with high sulfide content and high S/COD ratios, further research and development is needed to evaluate the performance of the IPSR while treating super-high sulfur wastewater. Such studies should determine the maximum feasible level that can be treated and improve gas stripping efficiency.

4. CONCLUSIONS

This study evaluated the performance of sulfide and COD removal by an IPSR. The results showed that the IPSR has unique mechanisms for H₂S removal before methane production occurs by using produced biogas. As a result, the IPSR had higher

organic removal than the control reactor. Additionally, the IPSR removed twice the H₂S of the control at a sulfide concentration of 400 mg S/L. Because of its high sulfide removal, the IPSR tolerated sulfide concentrations in the influent wastewater at high levels that caused the control reactor process to collapse.

ACKNOWLEDGEMENTS

We thank Mrs Naoko Okubo for support with the experiments. We thank Edanz (<https://jp.edanz.com/ac>) for editing a draft of this manuscript.

FUNDING

This study was supported by KAKENHI [grant number JP18K04416] from JSPS and the Steel Foundation for Environmental Protection Technology, Japan.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

T.O. and K.S. have patent #Japanese Patent No. 6029081 issued to Licensee.

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First received 19 December 2022; accepted in revised form 10 April 2023. Available online 18 April 2023