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Assessment of limited downstream nitrate dosing for sulphide control in pressure sewers: case study in Germany

Daneish Despot*, Luisa Reinhold and Matthias Barjenbruch

Institute of Civil Engineering, Chair of Urban Water Management, Technical University of Berlin, Sekr. TIB1 – B16, Gustav-Meyer-Allee 25, Berlin 13355, Germany

*Corresponding author. E-mail: daneish.despot@uwi.tu-berlin.de

DD, 0000-0002-8980-5651

ABSTRACT

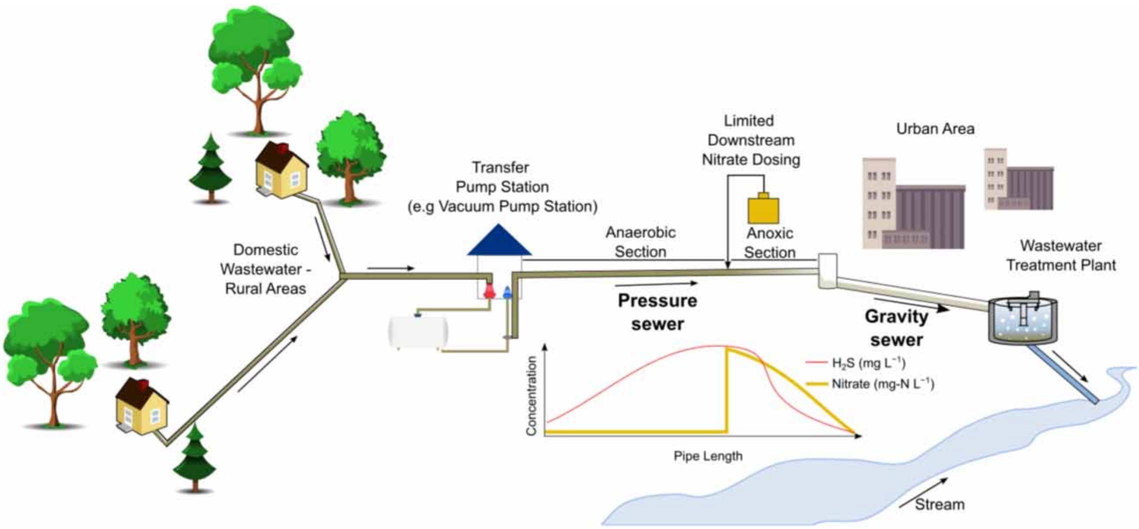
Odour release from sewer systems is an established environmental issue affecting many cities worldwide. A key indicator for the severity of the odour problem in sewer systems is linked to the amount of sulphide produced and released as hydrogen sulphide (H_2S) into the urban environment. For this reason, an in-depth understanding of the biochemical processes of sulphurous compounds in sewer systems is an underlying base point for designing and implementing odour control strategies for sewer systems. This contribution focuses on a field study on the monitoring and assessing chemical dosing on a 9.0 km pressure sewer in Ueckermünde, Germany. Online H_2S measurements indicated a daily reoccurrence of sulphide spikes between 4:00 and 8:00 am during the monitoring period. During this period, the wastewater slugs exiting the sewer section remained in the dosed section of the pipe the longest, and regeneration of sulphide production began after 3 hours, causing the hydrogen sulphide spikes. The proposed dosing strategy provides a cost-effective and efficient solution for sulphide build-up in long pressure sewers that connect rural areas to centralised wastewater collection and treatment systems.

Key words: nitrate dosing, odour and corrosion, pressure sewers, residence time, sulphide control

HIGHLIGHTS

- The sulphide problem in the studied sewer system is highly influenced by extremely long retention times leading to the failure of conventional dosing strategies.
- Using limited downstream nitrate dosing, 1.5–5 mg-N L^{-1} reduced the H_2S concentration to 0.60–0.26 mg L^{-1}.
- The applied dosing strategy provides an effective solution for controlling H_2S sewers typically used to connect rural areas to centralised systems.

GRAPHICAL ABSTRACT



1. INTRODUCTION

Wastewater discharges from intermittently operated pressure sewers used for collection services in rural and suburban areas are ubiquitously known to cause severe odour and corrosion problems. In central and eastern Europe, pressure sewers are commonly used to convey wastewater from rural and suburban areas to a centralised wastewater treatment plant. With the pressures of the European Water Framework directives in 2000 to expand the scope of water protection and achieve good chemical and ecological quality for all waters (surface and groundwater), the member state countries needed to rethink their sanitary services in their rural regions. In response to this mandate, extending the sewerage system was one option implemented by several water service providers (Gerend 2019). This transformation resulted in the installation of long pressure sewers to collect and transport wastewater in rural and suburban areas to a centralised system where the wastewater is directed to a treatment facility.

The wastewater transported in pressure sewers found in rural areas is typically characterised by long residence times and is under anaerobic conditions for most of the time. Under these conditions, sulphate-reducing bacteria (SRB) are active and play an important role in the sulphur and carbon cycle by coupling the production of hydrogen sulphide with the oxidation of organic compounds (Villahermosa *et al.* 2016). Sulphate reduction by the SRB residing in biofilms on the sewer walls is mainly responsible for the build-up of H_2S concentrations in the bulk wastewater (Li *et al.* 2017). H_2S is subsequently released to the gas phase at points in the sewer network associated with turbulent conditions and is influenced by the liquid phase H_2S concentration, pH and temperature. In the gas phase, H_2S is well-known for causing microbial-induced corrosion in concrete sewers, obnoxious odours, and its toxicity to sewer workers and residents when released to the sewer atmosphere and its surrounding areas.

To avoid corrosion, an H_2S concentration of less <3 ppm is necessary to mitigate corrosion risk to a rate of 10 mm a^{-1} (Weissenberger 2002), and odour is perceptible from 0.5 ppm H_2S (Hvitved-Jacobsen *et al.* 2013). To achieve these H_2S control targets, mitigation strategies aim to inhibit the production of sulphide by inactivating SRB bacteria, reducing the H_2S concentration in wastewater, or treating the H_2S gas released. However, the problems are often detected in 'already built' sewer systems, so construction measures to avoid odours are difficult to implement. Possible countermeasures in an existing sewer system include the introduction of oxidising compounds, e.g. hydrogen peroxide (H_2O_2) or oxygen (O_2) (Saračević 2009; Urban 2010), the dosing of alternative electron acceptors like nitrate, the precipitation of sulphides, e.g. by iron salts or the prevention of H_2S emission by increasing the pH level (ATV-DVWK 2003; Zhang *et al.* 2008).

In Germany, mainly calcium nitrate, flushing with pressurised air and iron salts are used (ATV-DVWK 2003; Ott 2004 in Barjenbruch 2007). A chemical dosing survey in Australia revealed that iron salts and oxygen are mainly used for sulphide control (Ganigue *et al.* 2011). They are easy to implement and cheap, and many companies specialise in implementing such dosing systems. The most straightforward implementation is to continuously dose the chemical into the wet-well (Feldhaus *et al.* 2005; Frey 2008). For both these strategies, chemical dosing is made at the upstream point. Therefore, enough chemical is required to prevent anaerobic conditions throughout the entire length of the pipe or precipitate the sulphide produced by the sewer biofilms. Hence, for the nitrate dosing, enough nitrate has to be added to account for the consumption in the wastewater, biofilms and sediments (Friedrich *et al.* 2004; Frey 2008). The disadvantages of nitrate dosing at an upstream point such as the wet-well are the consumption of easily degradable COD, the possible accumulation of polysulfides leading to higher sulphide production once the dosing is stopped, the adaptation of the denitrifying bacteria increasing the amount of chemical and thereby costs over time, a thickening of the biofilms and the possible stimulation of SRB downstream of the dosed sections (Feldhaus *et al.* 2005; Frey 2008; Mohanakrishnan *et al.* 2008; Saračević 2009; Auguet *et al.* 2015; Liang *et al.* 2016). Some of these problems might be reduced by a different dosing strategy: the downstream dosing of nitrate, as suggested in (Auguet *et al.* 2015) and in (Friedrich *et al.* 2004). In this approach, less nitrate is dosed. Only enough nitrate for oxidation of the sulphide that is already generated and the consumption in the remaining pipe section is required. Thereby chemicals can be saved, and the adaptation of biofilms is also reduced due to the limited amount dosed (Auguet *et al.* 2015).

This study aims to assess the effectiveness of limited downstream nitrate dosing for sulphide control on a 9.0 km pressure sewer pipe. Year-long monitoring of the liquid phase H_2S concentration was made to capture the seasonal effects in response to the downstream dosing strategy. Furthermore, in this study, we also assessed the impact of residence time on the performance of downstream nitrate dosing in an attempt to determine the period that was dosed ineffectively. As part of understanding the influence of the residence time on the dosing strategy, we examined how the pump operation schedule at the transfer station and hydraulic conditions could influence the sulphide formation, which can provide valuable insight into optimising the pumping schedule for other sewer systems with similar characteristics. Finally, we demonstrated how wastewater flow data could help optimise the dosing strategy and reduce chemical input. In the end, a cost comparison of upstream and downstream dosing is performed to showcase the value of applying a limited downstream nitrate dosing.

2. MATERIAL AND METHODS

2.1. Description of the study site and existing dosing applications

The small town of Ueckermünde is located at the eastern edge of Mecklenburg-West Pomerania, close to the Polish border, with approximately 9,000 inhabitants. Wastewater collection services extend to the rural villages west of Ueckermünde (Leopoldshagen, Mönkebude and Grambin). The wastewater from these villages is transported through two pressure sewer sections (Mönkebude and Grambin sewer section) which discharge into a gravity system that conveys the wastewater to the Wastewater Treatment Plant (WWTP). With the Baltic Sea being nearby, Ueckermünde is a popular tourist destination during the summer. However, in the last years, the increasing number of odour complaints by visitors and residents have led to the design and implementation of two chemical dosing stations to suppress the formation of hydrogen sulphide. These dosing stations were strategically installed to reduce the sulphide produced in each of the two main sewer sections. In reference to the wastewater flow (Figure 1), the first chemical dosing station is placed at the start of the pressure sewer, where flow-proportional dosages of ferrous chloride ($FeCl_2$) are administered. The second dosing unit is installed at a downstream point near the end of the pressure sewer, where a limited nitrate dosing strategy is applied.

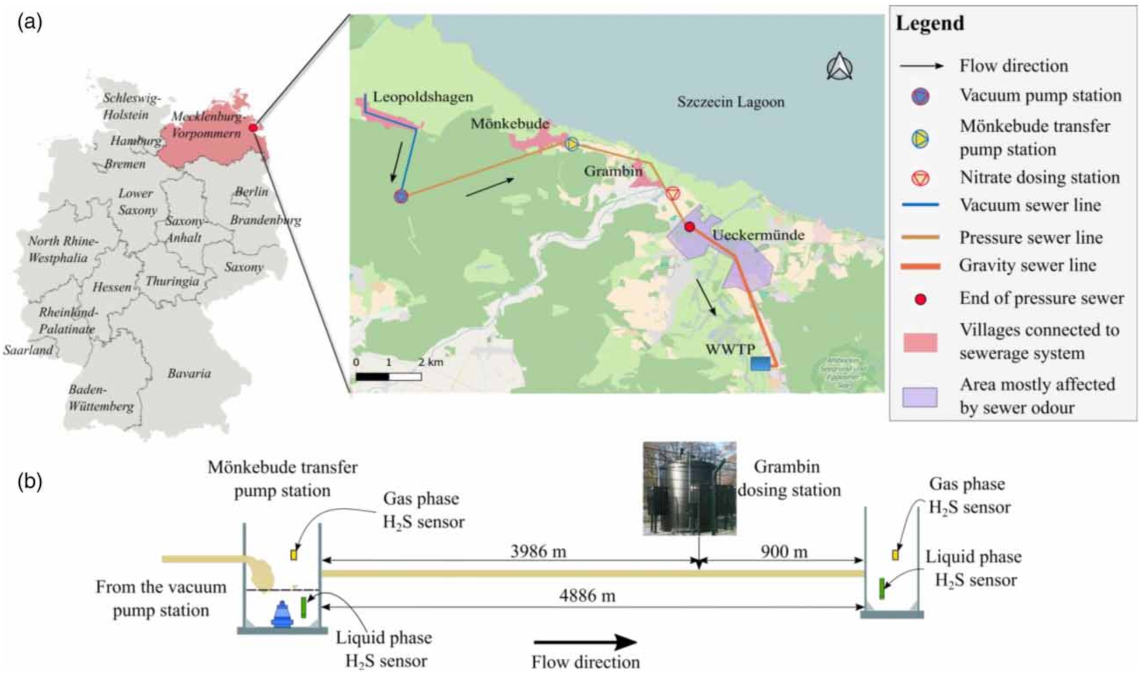


Figure 1 | Layout of the pressure sewer system used for wastewater collection in rural villages west of Ueckermünde (a) Grambin Sewer Section (GSS)—Mönkebude to the end of the pipe. (b) The Grambin Dosing Station (GDS) is installed 900 m before the end of the pipe. Continuous monitoring of hydrogen sulphide in the liquid phase from 11.10.2019 to 03.03.2020 using the SulfiLogger^{TM}.

At the first dosing station, installed at the Leopoldshagen vacuum pump station, $FeCl_2$ is used to suppress the sulphide produced in the Mönkebude sewer section (L = 4.9 km, DN = 200 mm) and thereby aims to alleviate the release of H_2S when discharged in the Mönkebude transfer station. One of the significant contributors to the sulphide problem of this sewer section is the abnormally long travel time through the pressure sewer system. Daily wastewater volumes ranging from 3 to 50 m^3 per day are periodically pumped into this section resulting in residence times ranging from 16 to 48 hours. Even with the ferrous chloride dosing of 66 L d^{-1} applied upstream, relatively high H_2S concentrations persist and are only partially effective in controlling the sulphide formation in this sewer section. Furthermore, the chemical dosing in this section is plagued with several operational challenges, for example, the formation of iron precipitates at the dosing point, which was assumed to affect the magnetic field of the wastewater IDM flow meter resulting in false wastewater flow. Consequently, this results in disproportional $FeCl_2$ concentrations (chemical delivery per volume of wastewater).

Given the challenges of the upstream ferrous chloride dosing and that there were ongoing plans to reconfigure the upstream dosing, we focus our efforts on reducing the sulphide concentrations in the Grambin sewer section (GSS). The wastewater from the Mönkebude sewer section is transferred into the GSS ($L = 4.9$ km, averaged $DN = 190$ mm) at the Mönkebude transfer pump station (Figure 1). At the discharged point, wastewater from the pressure pipe enters the gravity sewer, which runs through the populated areas in the town of Ueckermünde before making its way to the treatment plant. At this point, the wastewater has its first contact with air and due to its septic status, it causes serious odour problems along the first 200 m from the point of exit. Furthermore, the hydraulic residence time in GSS ranges from 8 to 20 h, depending on the time of day. To reduce the intense odours at the end of the pressure sewer and its surroundings, a chemical dosing station was installed near the downstream end of the pressure sewer section, as shown in Figure 1.

2.2. Flow and residence time analysis

The residence time analysis for the sewer section conveying the wastewater from the wet-well in Mönkebude towards the treatment plant is focused on answering three main questions: (1) What is the residence time of the wastewater slug when it arrives at the dosing station (DS) and at the end of the pressure pipe?; (2) How long the wastewater slug resides in the anoxic section of the pipe (the dosed sewer section before the end of the pipe)?; (3) To what extent do the residence times vary? According to Chen *et al.* (2014), the residence time (RT) of a wastewater slug is the time the slug takes to travel from the inlet to the outlet of a pipe. The wastewater slug is defined as an infinitely small volume of sewage, also referred to as packages or parcels of wastewater pumped in each wastewater pumping event. Using the dimensions (diameter and length) of the sewer section and assuming that sewage transport through the section follows the principle of an ideal plug-flow reactor, the RT of the sewage slug is the time when the accumulated sewage pumped into the pipe is equal to the total volume of the pipe (1):

$$V = \int_{t_0}^{t_0+RT} Q_{in}(t)dt \tag{1}$$

where V is the total volume of the sewer section, RT is the residence time, t_0 is the time when the sump volume enters the pipe and $Q_{in}(t)$ is the wastewater flow as a function of time.

Since no records of the pump's operation data (on and off time) at the transfer wet-well pump were available, we determined the start and stop of the pump by identifying the turning points (local minimum and maximum points) of the flowrate data. Figure 2 illustrates the concept of identifying the on and off times of the pump. Using the start and stop times based on the local minimum and maximum points, the time taken for the slug to arrive at the end of the pipe was calculated. A python script was written to automate this process and return the time at which the pipe volume was totally filled due to accumulated sewage from the subsequent pumping events. Finally, subtracting the time taken to arrive at the point of interest (dosing point or end of the pipe) from the pump start times for each wastewater slug revealed the residence time of that particular slug. Following this simple and novel procedure, the diurnal variation of the residence times of wastewater slugs passing through the GSS was analysed.

To identify the dry weather flows, we followed the guidelines in ATV-DVKW A 198E (Standardisation and Derivation of Dimensioning Values for Wastewater Facilities 2003). According to the standard, the daily dry weather flow can be determined either by using the number of dry days based on the weather log of wastewater treatment plants (Chap. 4.2.2.1 Para. 1) or by using the mathematical derivation based on Fuchs *et al.* (2003) (Chap. 4.2.2.1 Para. 4). Both methods above were applied in this study.

2.3. Dosing scheme

The principle idea of the limited downstream nitrate dosing strategy is to oxidise the sulphide produced upstream and ensure anoxic conditions in the downstream dosing point to prevent further sulphide build-up (Yuan *et al.*

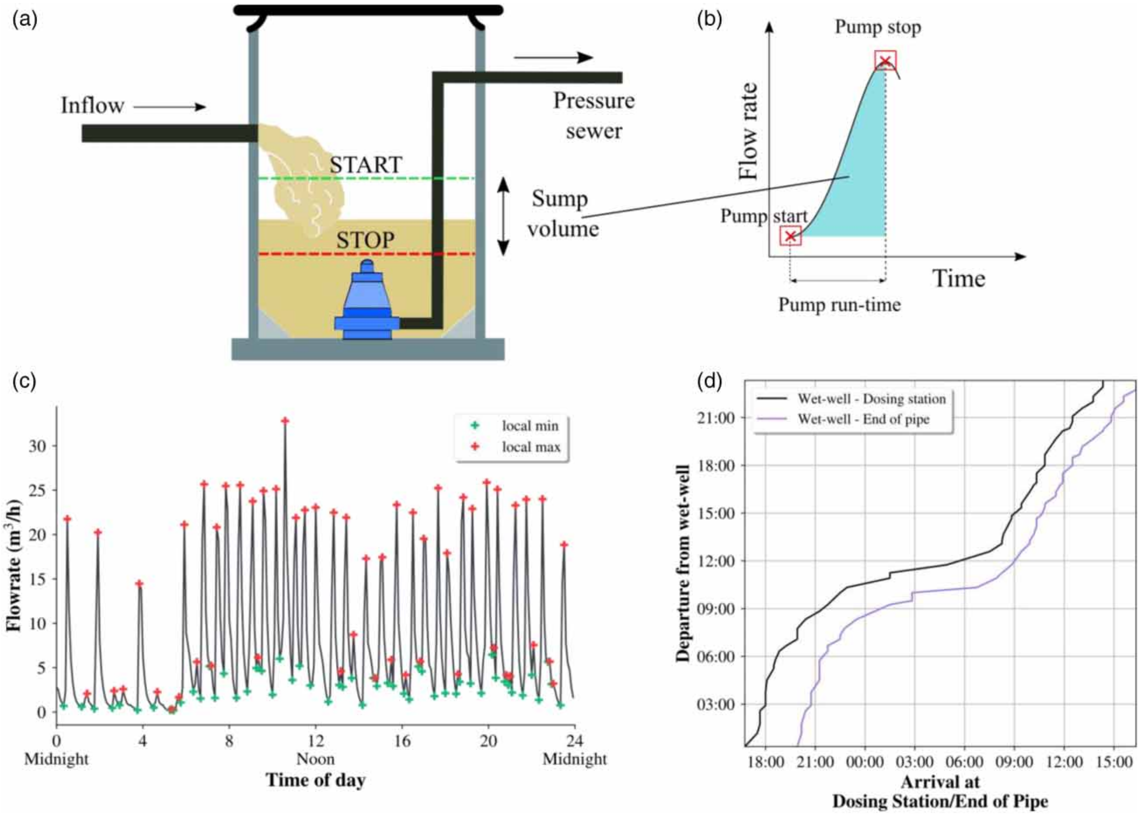


Figure 2 | Concept for calculating the residence times in the pressure sewer. An example flow rate time-series with identified start (local minima) and stop (local maxima) times (a,b). Illustration of selecting the pump on and off times for residence time computation (c). The sump volume pumped during the pump run-time is the wastewater slug or parcel entering the rising main at that given pumping event. Example of resulting departure/arrival time curves of wastewater slugs (d).

2015). The nitrate requirements for optimised dosing depend on two factors: (1) the amount of sulphide produced in the upstream section that needs to be oxidised; and (2) the nitrate that will be consumed for heterotrophic organic matter oxidation during the residence time in the dosed section, defined as the anoxic residence time (Yuan *et al.* 2015). We applied a limited nitrate supply during the monitoring period in which hydrogen sulphide (H_2S) concentration in wastewater was monitored to evaluate the dosing effectiveness under these conditions. With limited dosing, we prevent the nitrate-reducing bacteria established from consuming unnecessary amounts of nitrate. The amount of nitrate required for sulphide oxidation to sulphate is 0.7 g NO_3-N g^{-1}S-H_2S (2) (Yuan *et al.* 2015):

$$5H_2S + 8NO_3^- \rightarrow 5SO_4^{2-} + 4N_2 + 2H^+ + 4H_2O \tag{2}$$

The amount of sulphide produced in the anaerobic sewer section, until the DS was determined using Equation (3) (Hvitved-Jacobsen *et al.* 2013). Total dissolved sulphide measurements (DS_t: H_2S+HS^- also referred to as sulphide) made in 2018 and 2019 at the inlet ($C_{DS_t,\,inlet}$) and outlet ($C_{DS_t,\,EOP}$) of the Grambin sewer section for the periods when no chemical dosing were used to determine the area sulphide production of the biofilm (sulphide flux). The maximum sulphide flux ($r_{a,\,max}$) values measured for each season were used to estimate sulphide concentration produced until the wastewater slug arrived at the dosing point ($C_{DS_t,\,DS}$). We assumed that the sulphide flux at the end of the pipe was of a similar magnitude as at the dosing point and therefore used the anaerobic residence time (time taken for the wastewater slug to arrive at the dosing point, $t_{r,\,DS}$) and pipe dimensions (area: A, volume: V) to estimate the sulphide concentrations at the dosing point .The sulphide concentration at the dosing can be expressed by Equation (4). It is directly affected by the sulphide produced until the dosing point ($C_{DS_t,\,anaerobic}$) and $FeCl_2$ dosing on the Leopoldshagen sewer section which constitutes the wastewater entering the treated sewer section. To express the concentration as the unionised sulphide species, H_2S, at the dosing point, we converted the estimated $C_{DS_t,DS}$ for different seasons following (Despot *et al.* 2021). This value was then used to calculate the nitrate dosing rates for the different seasons. Table 1 summarises the dosing rates that were used in this study:

$$\Delta C_{DS_t} = C_{DS_t,EOP} - C_{DS_t,inlet} = r_{a,max}\frac{A}{V}t_{r,EOP} \tag{3}$$

$$C_{DS_t,DS} = C_{DS_t,anaerobic} + C_{DS_t,inlet} \tag{4}$$

where:

ΔC_{H_2S}: H_2S produced when the wastewater slug arrives at the end of the sewer section.

$t_{r,EOP}$: residence time of the wastewater slug when it arrives at the end of the sewer section.

Table 1 | Seasonal nitrate dosing rates applied to Grambin dosing station

	Estimated H_2S[a] (mg-S L^{-1})	Nitrate (mg-N L^{-1})	kg $Ca(NO_3)_2$ solution d^{-1}	L $Ca(NO_3)_2$ solution m^{-3}
Winter	5.51	3.90	10.19	0.035
Spring and autumn	7.00	4.90	12.93	0.045
Summer	8.29	5.80	15.31	0.053

[a]Estimated H_2S based on the maximum area sulphide production rate and residence time of wastewater slug arriving at the dosing point.

Flow proportional nitrate addition is made by a Memdos LP 4 (Lutz-Jesco GmbH) dosing pump which is operated in analogue input mode. At maximum operating capacity, the dosing pump can dose up to 4 L h^{-1}. When operated in analogue input mode, an external 0/4–20 mA signal controls the stroke frequency of the dosing pump. In this case, the external signal comes from the non-invasive ultrasonic flow meter (FLUXUS F501, SebaKMT), in which a wastewater flow rate of 90 m^3 h^{-1} is used as the current value (20 mA) for 100% delivery capacity whereas, for 0% delivery capacity, a current value of 4 mA is used for a flow rate of 0 m^3 h^{-1}. Based on the measurements obtained from the 2018 to 2019 monitoring campaign, the amount of $Ca(NO_3)_2$ solution delivered per cubic meter of wastewater is indicated in Table 1.

2.4. Evaluation of chemical dosing

For the evaluation of the chemical dosing trials, we calculated the H_2S removal efficiency of the dosing and classified the H_2S measurements under dosed conditions according to defined evaluation criteria. Baseline periods (reference period where no chemical dosing was administered) were established by switching off the chemical dosing pumps. Periods selected for baseline monitoring (without dosing) lasted for 5–8 days, with the first two days not included in the evaluation dataset. Leaving out the first 2 days ensured that the nitrate residuals in the dosed section were completely flushed out. The evaluation criteria were based on four risk levels for sewer corrosion and odour. The risk is associated with the measured H_2S concentration and is grouped into either negligible, medium-low, medium-high, or high (CH2M 2017). The evaluation criteria for classifying the H_2S measurements into respective risk groups are presented in Table 2.

Table 2 | Target concentrations of H_2S in the liquid and gas phase for evaluation of chemical dosing (CH2M 2017)

Colour	H_2S liquid phase concentration (mg-S L^{-1})	H_2S gas phase concentration (ppm)	Corrosion rate (mm/year)	Odour/corrosion risk	Comments
Green	0–0.5	0–5	$<=1$	Negligible	Typical civil assumptions for sewer corrosion rates
Yellow	>0.5–2	>5–20	>1–2	Low/Medium-Low	Typical average sewer profile
Orange	>2–4	>20–40	>2–4	Medium/Medium-High	Intervention likely to be in the near future
Red	>4	>40	>4	High	Potential issues of widespread, urgent action may be required

The unionised form of sulphide, H_2S (referred to as liquid phase H_2S in this article), was the chosen parameter for optimisation of the downstream nitrate strategy applied in this study. We chose this parameter because (1) only the unionised H_2S can be emitted to the sewer atmosphere, causing odour and corrosion problems; (2) the sensor used for optimisation directly measures the unionised H_2S. The SulfiLogger^{TM} S1/X1-1020 (SulfiLogger A/S, here forth referred to as SulfiLogger^{TM}) were installed in the Mönkebude transfer station (inlet) and at the end of the Grambin sewer section (Figure 1) for monitoring the liquid phase H_2S. Measurements were made in 1-minute intervals between 11.10.2019 and 03.03.2020 using the SulfiLogger^{TM}.

To characterise the activity of the SRB during the baseline monitoring periods, the liquid phase H_2S measurements were converted to total dissolved sulphide concentration following (Despot *et al.* 2021). Since the pH values recorded during the baseline monitoring were not made continuously, the median of measurements obtained was used in the conversion procedure.

2.5. Wastewater sampling and analysis

Grab samples were taken at the inlet and outlet of the treated sewer section during the baseline and chemical dosing monitoring field campaigns. The samples were immediately stored in a cooler box and taken to the laboratory, where they were either measured immediately or cooled to 4 °C and analysed the next day. For the sulphide measurements, 250 ml of the sample was preserved on-site by adding aluminium and sodium hydroxide for flocculation and zinc acetate to avoid losses during transportation to the lab. Total dissolved sulphide measurements were made using a modified version of Saračević (2009) with the sample preservation method adapted from the APHA Standards (APHA 2018). Temperature and pH measured on-site using HQ-D40 electrodes (Hach-Lange) were used for conversion between the sulphide species. For the soluble COD, sulphate and nitrate measurements, cuvette tests by Hach-Lange were used (LCK 514, LCK 153, LCK 339).

3. RESULTS AND DISCUSSION

3.1. Wastewater and baseline monitoring

To understand the wastewater composition entering the pressure sewer and its subsequent transformations relative to the sulphide build-up in pressure mains, sulphide and sulphate, COD, dissolved oxygen, pH and temperature were monitored both at the inlet and outlet of the pressure sewer. The wastewater composition at the inlet of the pressure sewer can be described as medium strength (Hvitved-Jacobsen *et al.* 2013). At the inlet (Mönkebude transfer station), the soluble COD ranged from 373 to 413 mg L^{-1}, with the highest values being measured during the winter. Generally, the soluble COD for this study site accounted for 40% of the total COD. Sulphate concentrations ranged from 62 to 183 mg SO_4^{2-} L^{-1}, with the highest values, also detected in winter. The pH values measured for the different seasons were in a similar range, between 7.8 (winter) and 8.3 (summer). Regarding the sewage temperature at the study site, a 10 °C difference between summer and winter temperatures was recorded. Differences between the inlet and outlet sewage temperature were minor (see Figure S2). Under dry weather flow conditions, the dissolved oxygen concentration in both the inlet and outlet of the sewer section was $<$1 mg L^{-1}.

Typical diurnal liquid phase H_2S profiles at the inlet and outlet of the pressure sewer when dosing was switched off are shown in Figure 3(a) (typical gas and liquid H_2S profiles and measurement distribution at the outlet of the Grambin sewer section are shown in Figure S1). The times associated with pumping intervals of the transfer pump station are shown to highlight the frequency of pumping events during the day. The periods of low pumping events (2:00 am to 5:00 am) correspond to the lowest sulphide concentrations at the outlet. During this time, there is no flow out of the pressure sewer. The wastewater is stagnant and remains in the same position for several hours before the incoming wastewater parcel displaces it. The wastewater temperature recorded during these periods is the lowest since the wastewater is exposed to colder night temperatures for several hours. As measurements are made in manholes and the wastewater parcel being measured is stagnant in the early morning periods, the overlying headspace facilitates the transfer of oxygen into wastewater, promoting sulphide oxidation and ultimately reducing the sulphide concentration. At the same time, SRB activity is hindered due to mass transfer limitations under quiescent conditions. These processes contribute to the drop in sulphide concentration during these periods.

Using the calculated anaerobic residence time and the area to volume ratio of 40 m^{-1}, the maximum area sulphide production rate of the biofilm was calculated to be between 0.43 and 2.1 g S m^{-2} d^{-1} for the baseline monitoring periods of the different seasons (Figure 3(b)). The sulphide production rates measured during

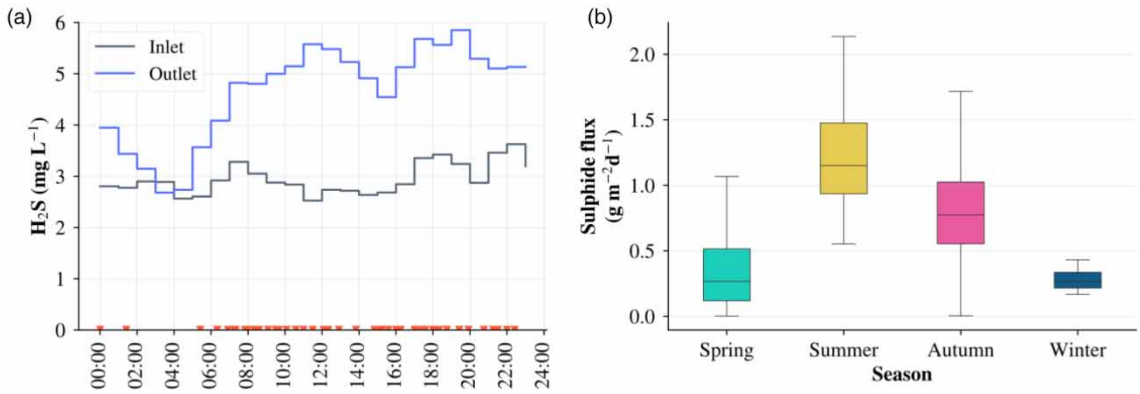


Figure 3 | Daily dissolved sulphide concentrations in the liquid phase in the inlet and outlet of the pressure sewer connecting Mönkebude and Ueckermünde (a) and average sulphide concentrations for the varying seasons (b). All measurements shown for baseline monitoring without dosing.

summer had the highest values, which coincides with a temperature increase of 10 °C in reference to the wastewater temperature in the winter. Therefore, the seasonal variation in the sulphide production rates indicates the changing SRB activity throughout the year and the need to implement a season-factor dosing scheme. It is important to realise that the transition periods from winter to spring and from summer to autumn may consequently result in periods of underdosing and overdosing and, therefore, should be carefully investigated. The effects caused by the transition periods were observed in our study, with noticeable differences between the area sulphide production rates in autumn and spring. Finally, it is important to note that sulphide formation persists during the colder months of the year. Consequently, it is necessary to implement chemical dosing throughout the whole year.

3.2. Flow and residence time analysis in dosed sewer section

Only dry weather flows were considered for the chemical dosing optimisation procedure. During wet weather flows, the effects on the chemical and microbial processes are minimal, mainly due to wastewater dilution (up to 50%) and the shortened residence times in the sewers because of increased wastewater capacity and pumping frequency. For this reason, we primarily focused on assessing the residence time attributed to dry weather flows. Dry and wet weather flows were identified using the ATV-DVKW A 198E guidelines (Standardisation and Derivation of Dimensioning Values for Wastewater Facilities 2003) (Figure S3). At this study site, dry weather flows occur 60% of the time, with the highest dry-to-wet ratio in the autumn (1.8) and the lowest during the summer (1.2). The dry weather flows and their corresponding anaerobic and anoxic residence times are shown in Figure 4(d) and 4(c). During the summer, the flow is, on average, around 20% higher compared to the average of all the other seasons. Because Ueckermünde is a popular tourist destination during the summer, there is an increase in the wastewater volumes, which explains the higher dry flows during the summer.

Figure 4(c) shows the anaerobic residence time, which corresponds to the time the wastewater slugs spend in the section from the Mönkebude transfer station to the Grambin DS. The anoxic residence times, shown in Figure 4(d), represent the duration the wastewater slugs stay in the anoxic or dosed section of the pressure sewer main from the DS to the end of the pressure pipe at the entrance to Ueckermünde town. It is visible that the anoxic residence time follows a distinct daily pattern. The slugs arriving at the end of the pressure sewer between 4:00 and 6:00 am have the longest travel time. Also, a clear pattern with the highest retention times between 5:00 and 7:00 am is visible for the anaerobic residence time, although it is not as defined as the anoxic retention time pattern. A reason for that could be the undefined pressure sewer connections from small pump stations and houses on the way from Mönkebude to Grambin. In contrast, there are no additional connections in the last part of the sewer. The lowest absolute residence time for both anoxic and anaerobic sections can be observed in summer and spring 2020, where stay-at-home measures due to the COVID-19 pandemic were in place. The reason for this is the higher flow in summer and during the first month of COVID-19 lockdown, shown in Figure 4(a) and 4(b). Abu-Bakar *et al.* (2021) and Lüdtke *et al.* (2021) demonstrated how the UK and Germany's COVID-19 lockdown measures drastically altered water consumption patterns. Both studies indicated that the lockdown measures instigated a significant increase in water consumption, directly translating

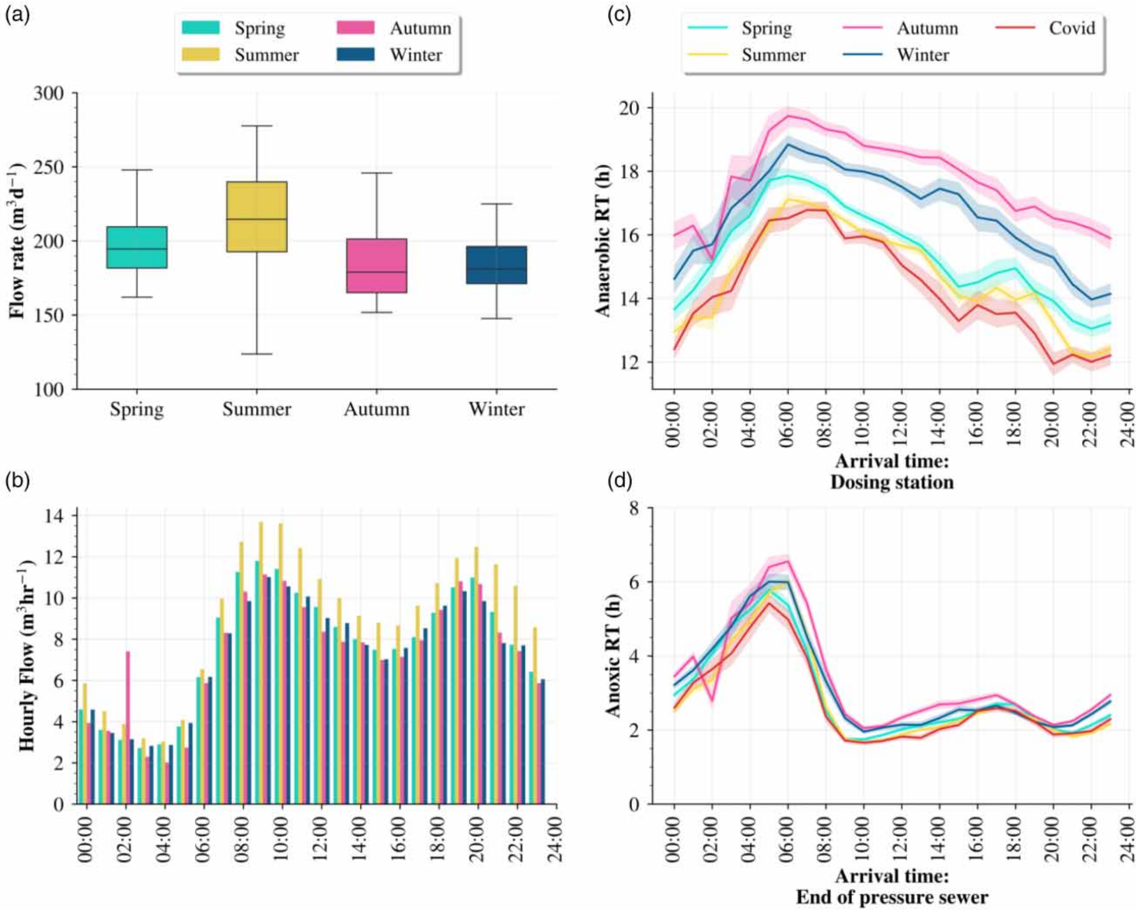


Figure 4 | Average daily flow rates for different seasons. (a) Diurnal profile of hourly flow for different seasons. (b) Diurnal profile of anaerobic residence time in the pressure pipe. (c) Diurnal profile of anoxic residence time. (d) Only dry weather flows are included.

into increased wastewater production. The lowest flow in autumn corresponds to the longest retention times in the anaerobic and anoxic sections.

Table 3 shows the summary of the residence time analysis. Considering both wet and dry weather flows, the mean daily flow of the Grambin sewer section is 191 m^3 d^{-1}. Although the sewer system is a separate sewer system, excess water during wet weather events increases the mean daily flow to 214 m^3 d^{-1}. The higher flow

Table 3 | Summary of average retention times in different seasons and weather conditions

	Daily flow Q_d (m^3 d^{-1})	Avg. pump events d^{-1}	Mean RT for arrival at GDS (h)	Mean RT in anoxic section, (h)	Mean RT for arrival at EOP (h)
Dry weather flow (DWF) (n=406)	177.36	59	14.3±3.3	3.1±2.1	17.4±3.6
Summer DWF (n=98)	202	54	13.2±2.9	3.2±3.0	16.4±3.2
DWF, all seasons except summer (n=308)	169	60	14.6±3.4	3.1±1.7	17.7±3.7
DWF_{high} 24.07.2019 (n=1)[a]	241	50	10.5±1.9	2.6±1.4	13.2±2.1
$DWF_{typical}$ 25.10.2019 (n=1)[a]	175	54	15.4±2.1	2.6±1.3	18.1±1.8
Wet weather flows only (280)	214	51	12.8±2.9	2.6±1.0	15.5±3.0
Storm 11.07.2018 (n=1)[a]	329	61	9.5±2.9	2.1±0.9	11.7±3.3
All flows (n=686)	191	56	13.6±3.2	2.9±1.7	16.5±3.5

RT, Residence Time; EOP, End of pipe; GDS, Grambin Dosing Station.
[a]Based on hourly mean values.

directly leads to a lower retention time. On average, the wastewater spends 16.5 h in the pressure sewer from Mönkebude to Grambin, the last 3 hours under anoxic conditions in the dosed section of the pipe. The residence times in our study are relatively long when compared to other field studies, which recorded highly septic wastewater after more than 3–7 hours (Frey 2008; Jiang *et al.* 2013a). With downstream nitrate dosing, an ideal residence time in the dosed section of less than 90 min is recommended due to the depletion of the oxidising agent and continuation of sulphide formation (Friedrich *et al.* 2004). The 3 hours recorded in our study are therefore longer than recommended for this dosing strategy. The residence time is dictated by the daily wastewater flow, which depends on the water consumption. Adapting the chemical dosing based on the system characteristics (defined by the consumption and storage capacity) must be considered when planning chemical dosing measures for sulphide control.

3.3. Effectiveness of the downstream nitrate dosing

To analyse the effects of the downstream nitrate dosing application, the mean H_2S measurements recorded under dosed conditions were computed for each season and compared to the baseline monitoring period. The H_2S diurnal variation was plotted to highlight the fluctuations of the H_2S with respect to the residence of the wastewater slugs (Figure 5). An H_2S removal efficiency of 86 and 84% was achieved for autumn and spring, respectively, using the current downstream-limited nitrate dosing application. The reduction target of <0.5 mg L^{-1} was met for 89% of the time during spring, as reflected in Figure 5(c). During the winter, 79% of the measurements had H_2S concentrations <0.5 mg L^{-1}, whereas only 56% for the autumn measurement period. 40% of the autumn measurements fell in the medium-low (>0.5–2 mg L^{-1}) risk group, indicating that corrosion rates of >1–2 mm per year are likely with mild odour problems. The desired result of achieving low H_2S during the spring is solely based on the improved performance of the $FeCl_2$ dosing during the periods of reduced residence time in the Leopoldshagen sewer section, which corresponded to the increased wastewater volumes during the COVID-19 lockdown measures.

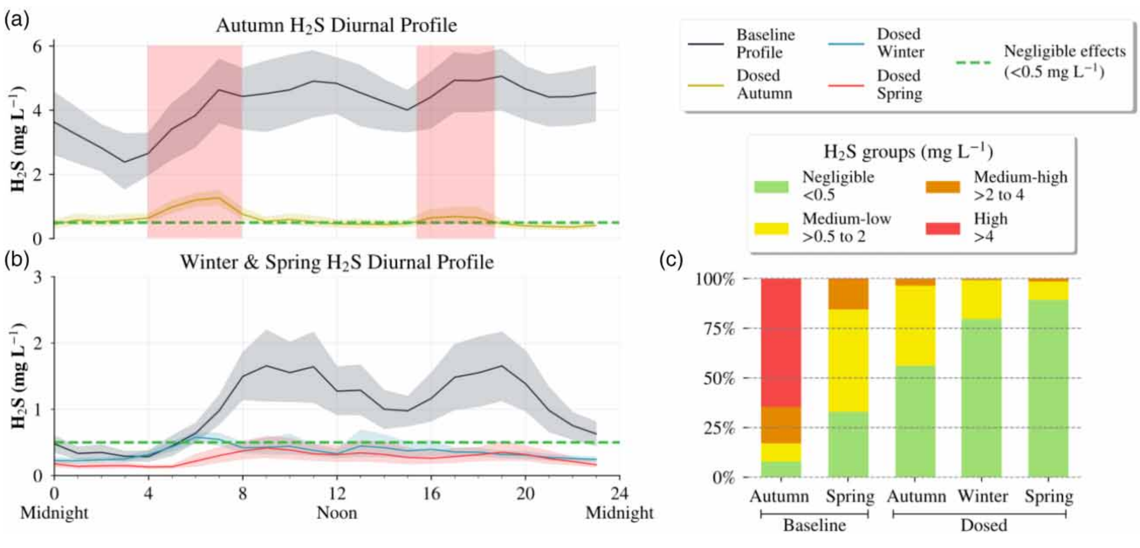


Figure 5 | Diurnal profile of total dissolved sulphide in the liquid phase for autumn, winter and spring (a, b) and the evaluation based on the odour and corrosion risk (c). Baseline profiles and their variation are shown in black (a, b).

The inability to achieve the reduction target during the autumn and winter monitoring periods is the result of the higher upstream H_2S loads resulting from the insufficient sulphide removal of the upstream $FeCl_2$ dosing. It was found that when the upstream sulphide concentration was less than 0.5 mg L^{-1}, the H_2S measured at the end of the pipe was <0.2 mg L^{-1}. Furthermore, the long residence times in the anoxic section promote the regeneration of sulphide production (Friedrich *et al.* 2004). When the nitrate added is utilised for the oxidation of the sulphide load coming from the wet-well as well as the sulphide generated in the anaerobic section before the dosing point, the residual nitrate falls to the typical nitrate concentration in domestic wastewater (<1 mg NO_3^--N L^{-1}). At this point, the artificial anoxic conditions no longer prevail, and SRB activity resumes. The sulphide oxidation process occurs within the first 2 hours after being dosed (Friedrich *et al.* 2004). It is assumed that the wastewater slugs are exposed to a transition from anoxic to anaerobic conditions between the 2nd to 3rd hour after entering the dosed section. The nitrate concentration was 0.8 mg NO_3^--N $L^{-1}\pm0.1$ (n=14) after the dosing was switched off for one day, which is typical for wastewater in pressure sewers. During dosing, the nitrate concentration downstream was, on average, 1.7 mg NO_3^--N $L^{-1}\pm0.3$ (n=8). These samples were taken between 9:30 and 11:00 am for technical reasons and do not represent the critical areas in the early morning where nitrate is completely depleted (see red shaded regions in Figure 5(a)).

When a residence time of 3 hours is exceeded, anaerobic conditions prevail once more, favouring the regeneration of sulphide in the pressure main. This effect is shown in the shaded regions (in red) in Figure 5(a), where the SulfiLogger™ was able to detect the regeneration of the sulphide because of the long residence times in the anoxic section. Another essential point contributing to the sulphide peaks highlighted in Figure 5(a) and 5(b) is the increasing residence times of the wastewater slugs arriving at the dosing point. At this time of the day, the residence times of the wastewater slugs approach their maximum, thereby increasing the sulphide concentration beyond the expected limit. This indicates that an additional amount of nitrate is needed to effectively reduce the sulphide concentration of the slugs arriving at the dosing point between 4:00 and 8:00 am. Furthermore, since the nitrate is also used as an electron acceptor by heterotrophic bacteria to oxidise organic matter, supplying an additional amount of nitrate for this process must be considered (Yuan *et al.* 2015).

3.4. Influence of residence time

Correlation plots of anoxic residence times and the measured H_2S at the end of the pressure main were made to examine the relationship between these two parameters. Figure 6(c) exemplifies a day with typical dry weather flow to highlight a fairly strong correlation (R^2=0.63) between the residence time in the anoxic section and the H_2S measured at the end of the pipe (Figure 6(c)). The diurnal sulphide profile for the autumn H_2S

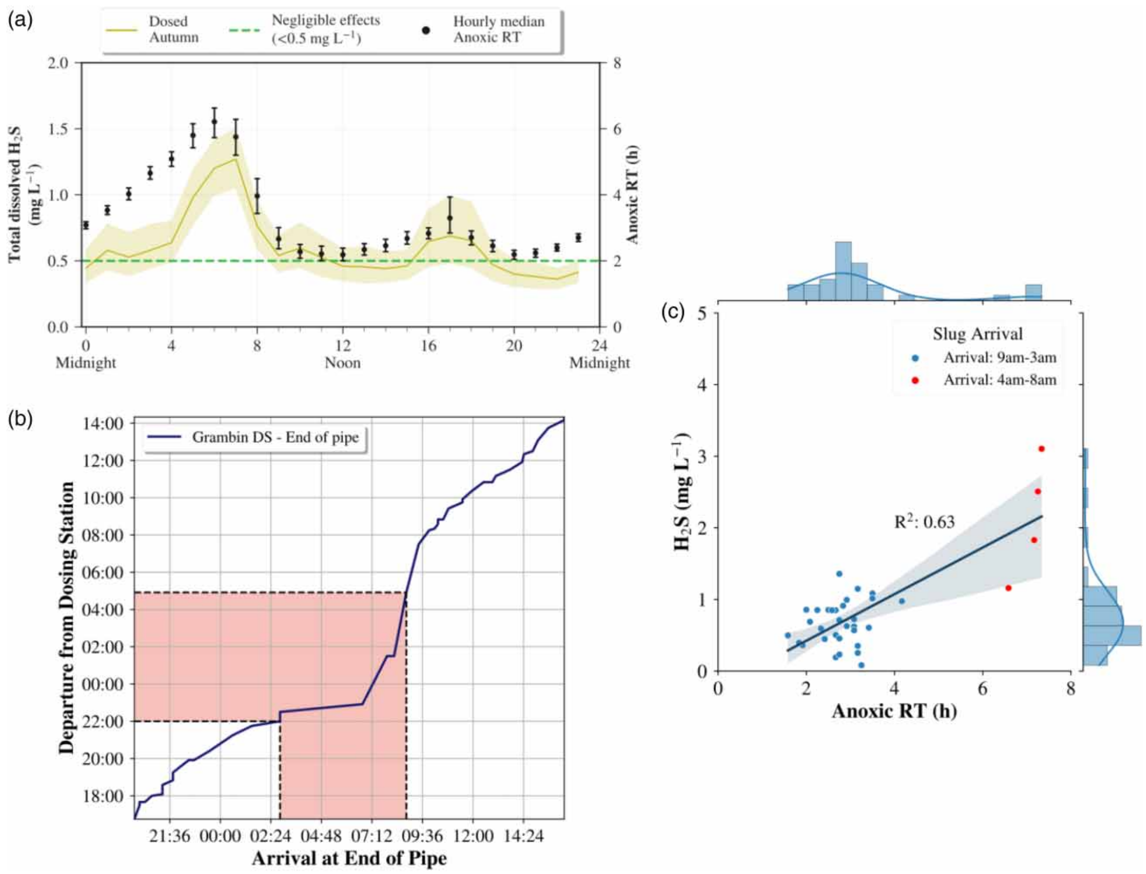


Figure 6 | Influence of anoxic residence time on the dosing effectiveness. Typical diurnal profile of total dissolved sulphide in the liquid phase. (a) Departure and arrival diagram for the respective wastewater slugs. (b) Correlation between anoxic residence time and the measured H_2S concentration in the liquid phase (c).

measurement mirrors the profile of the anoxic residence times, highlighting the strong influence of anoxic residence time on the sulphide peaks. Daily, two pronounced sulphide peaks were identified, one in the morning between 4:00 and 8:00 am and the other in the evening between 3:00 and 7:00 pm (Figure 6(a)). In Figure 6(c), we highlighted the H_2S measurements of the wastewater slugs arriving between 4:00 and 8:00 am in red to emphasise the higher sulphide concentrations during this period and that there are reoccurrences of higher H_2S concentrations during the period.

Figure 6(b) displays a typical departure–arrival time curve for the wastewater parcels transported through the dosed sewer section. Considering the interquartile range, departure times that had anoxic residence times >3 hours and corresponded to higher H_2S concentrations were identified to be in the time window between 9:30 p.m. and 3:30 a.m. This time window marked the arrival of the wastewater parcels from the Mönkebude pump station, which was likely to cause sulphide peaks between 4:00 and 8:00 a.m. Therefore, targeting the corresponding wastewater slugs entering the dosing point will improve the limited downstream nitrate dosing performance.

3.5. Further optimisation and recommendations

So far, the limited downstream nitrate dosing application has proven to be very effective given the low chemical cost per year (1,853 € yr^{-1}) and relatively high reduction percentages. A nitrate dosing range of 1.5–5 mg-N L^{-1} was shown to reduce the H_2S concentration to 0.60–0.26 mg L^{-1}. Despite the remarkable performance of the limited dosing strategy, we found that the success of the dosing at this point was strongly dependent on the residual sulphide concentration in the upstream wet-well, the sulphide build-up in the sewer section before the dosing point and the residence time in the dosed section. The anaerobic residence times of the wastewater slugs entering the dosing point are strongly influenced by the sulphide build-up in the un-dosed section and are responsible for the sulphide peaks occurring between 4:00 and 8:00 am. Moreover, when the residence times in the dosed section exceeded 3 hours, sulphide production resumed, and higher H_2S concentrations were detected by the SulfiLogger™.

To address the concerns of the upstream sulphide concentration and the wastewater slugs remaining in the dosed section for periods >3 hours, we propose the following measures:

1. Adjust the amount of nitrate added to account for upstream sulphide loads.
2. Implement residence-time-based dosing by defining the time window for the wastewater slugs residing in the anoxic section for >3 hours, targeting the sulphide peaks typically produced during 4:00–8:00 a.m.

To address the problem of the sulphide peaks between 4:00 and 8:00 a.m., we propose using a residence time-based dosing concept as shown in Figure 7. This dosing scheme aims to increase the dosage for the wastewater slugs with higher residence times, which generally has a higher sulphide concentration. Also, since the

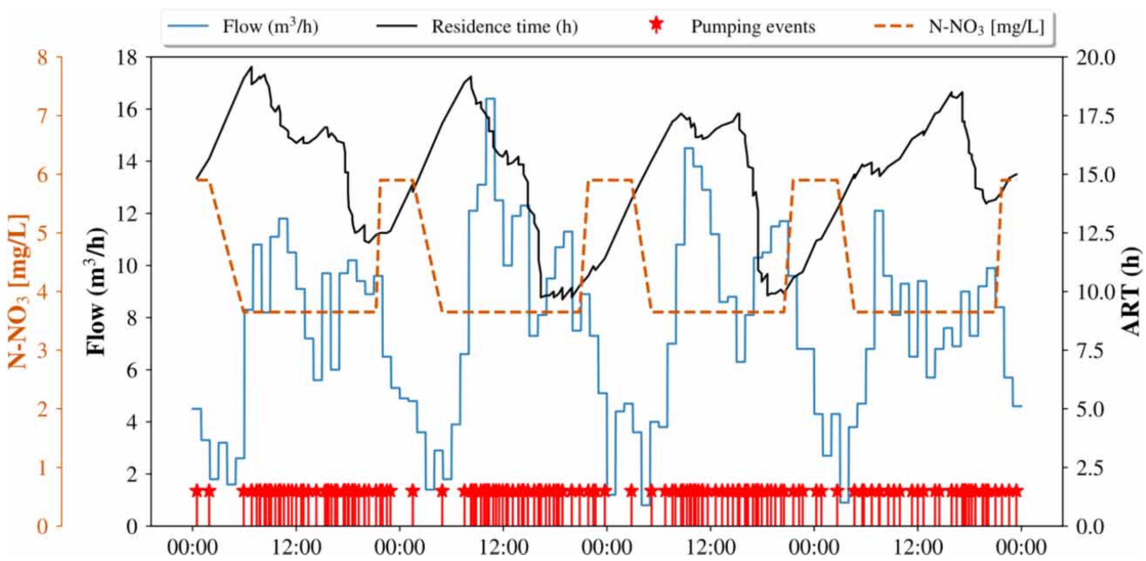


Figure 7 | Proposed nitrate dosing scheme shown together with flow, residence time and pumping events.

regeneration of sulphide in the dosed section occurred for residence times mostly >3 hours, we estimated the nitrate requirements for the sulphide produced when the residence times in the dosed section are >3 hours. Since the wastewater slugs arriving between 9:30 p.m. and 3:00 a.m. correspond to both these occurrences, we recommend raising the nitrate concentration by around 2.25 mg-N L^{-1} for this period. For calculating the sulphide regeneration, we used a biofilm sulphide production flux of 0.03 g m^{-2} h^{-1}, which is slightly higher than the ones used for the un-dosed section. The reason for this is the competitive nature of the sulphate-reducing bacteria and the established nitrate-reducing-sulphide oxidising bacteria (NR-SOB) in the dosed section, which triggers higher SRB activity (Jiang *et al.* 2013b).

3.6. Economic evaluation

For the economic evaluation, the theoretical cost of an upstream nitrate dosing scheme was compared to the nitrate requirements using the downstream nitrate dosing performed in this study and the scenario considering the further optimisation of the current nitrate dosing. The theoretical upstream nitrate requirements were made following (Yang & Hobson 2005) (further details can be found in the supplementary material – Theoretical upstream dosing calculation). The nitrate solution needed per day for the different seasons and the resulting yearly costs is shown in Figure 8. The values for the calculation can be found in Table S2. For the calcium nitrate solution cost, a price of 0.43 € kg^{-1} was assumed based on a nitrate product of the company VTA Austria GmbH.

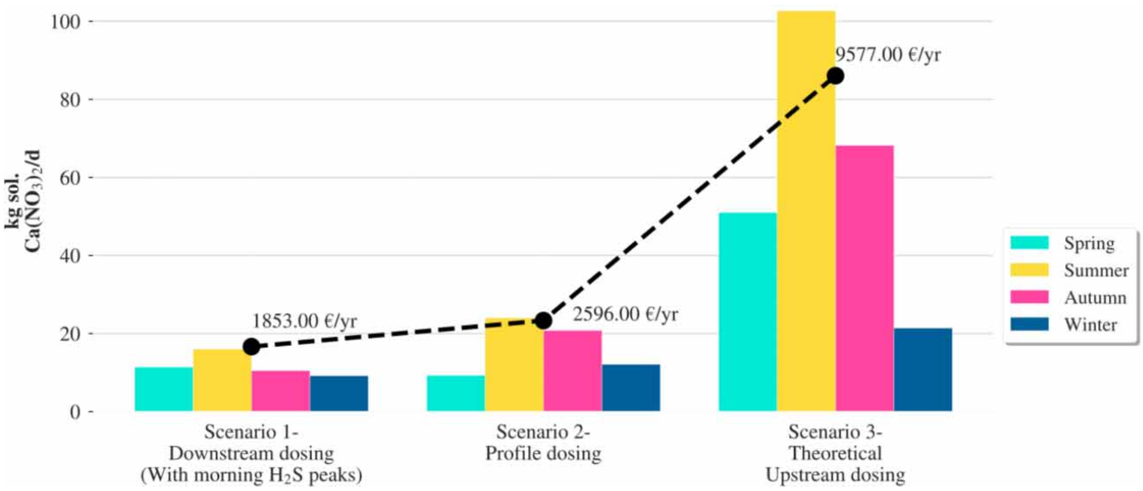


Figure 8 | Cost evaluation for the downstream dosing, the optimised downstream profile dosing and the theoretical cost for upstream nitrate dosing based on sulphide production calculations for the bulk wastewater and biofilms with average retention times and the resulting nitrate consumption.

For the upstream dosing, approximately five times more nitrate solution is needed than the downstream dosing as it is applied currently, hence almost five times the costs for chemicals. With the current dosing, sulphide peaks in the morning are still noticeable. However, the suggested profile dosing addresses the sulphide peaks (4:00–8:00 a.m.) only at an operating cost of around 750 € more per year. One reason is that the time window that requires higher nitrate dosages is the period of the lowest flow. The main reason for explaining the difference between the applied downstream dosing (Scenario 1) and the profile dosing (Scenario 2) is the difference in sulphide production rates between autumn and spring. Indeed the difference is a direct result of the autumn having higher residence times than spring. Furthermore, we used a slightly higher $N\text{-}NO_3/H_2S$ ratio to compensate for the consumption of heterotrophic organic matter oxidation during the transport in the dosed section (Yuan *et al.* 2015). It is important to emphasise that no special or additional equipment is needed for implementing Scenario 2 since the current dosing pump in Grambin can deliver different dosing rates considering a pre-defined pattern.

The nitrate demand is reduced by 73% when considering Scenario 2, which is expected to result in the complete abatement of H_2S produced in the Grambin sewer section. Similarly, Gutierrez *et al.* (2010) showed that nitrate consumption was reduced by 42% when a modified RT-based dynamic downstream dosing was performed on a sewer laboratory reactor. Overall, both limited downstream nitrate strategies (Scenario 1 and Scenario 2) offer potential savings and are significantly cheaper when compared to the theoretical upstream requirements.

According to Mohanakrishnan *et al.* (2009), upstream nitrate dosing increases the sulphide production capability of the downstream biofilm; this, in turn, increases the overall capability of the sewer section for sulphide production when anoxic conditions no longer prevail. If upstream nitrate dosing continues over time, higher nitrate dosages will be required, increasing chemical costs. The upstream nitrate dosing becomes increasingly expensive in the long run and for long pressure sewers. These findings should be considered when implementing a new dosing concept in any location where long pressure sewers are utilised.

3.7. Practical implications

Regarding undesirable effects of nitrate, especially when dosed in excess, nitrous oxide (N_2O) formation and emissions, especially if trace amounts of oxygen are present, are almost certain to occur (Zhang *et al.* 2021). N_2O is a severe greenhouse gas with a global warming potential of 298 times the one of CO_2 (US EPA 2019). According to Short *et al.* (2014), past research shows that the wastewater type (e.g., C/N ratio) and organic loading rate, aeration regime (aerobic–anoxic cycling) and environmental parameters such as dissolved oxygen (DO) concentration or pH influences N_2O generation in wastewater systems. In addition, the concentration of substrates (NH_4^+-N, NO_3-N) and intermediates (NO_2-N, NO and free nitrous acid), as well as the abundance and activity of N_2O-producing microorganisms (i.e. nitrifying and denitrifying microbes), are also linked to N_2O generation. Therefore, minimising the amount of the residual nitrate concentration of the dosed sewer sections is an important measure that must be considered when using N-based salts such as nitrate. Auguet *et al.* (2015) indicated that limited nitrate dosing downstream produces negligible N_2O emissions since the added nitrate was mostly utilised. However, in the case of upstream nitrate dosing, excess dosing is likely to result in high residual nitrate concentrations; therefore, N_2O formation is more likely. To evaluate the depletion of nitrate, nitrate was measured at the end of the pressure sewer, with the majority of the nitrate measurements (85%) having a concentration <1 mg N L^{-1}, indicating that added nitrate was mostly depleted. Also, the wastewater exiting the dosed sewer section is expected to be mixed and diluted with wastewater entering the main gravity sewer system at downstream points, further decreasing the possibility of N_2O formation.

Besides the possibility of contributing to the GHG, high nitrate dosing rates increase the consumption of readily biodegradable carbon sources during denitrification, which is likely to affect biological processes at the treatment plant (Liu *et al.* 2015). Furthermore, nitrate, among other oxidants like ferric iron and hydrogen peroxide, was found to cause the production of other volatile odorous sulphur compounds like dimethyl trisulfide when dosed intermittently (Gu *et al.* 2019). Furthermore, the presence of nitrate increased sulphide production compared to the original state when dosing is interrupted due to polysulphide accumulation during dosing (Liang *et al.* 2016).

According to the findings of Mathioudakis & Aivasidis (2009), temperature significantly impacts the denitrification kinetic profile. The percentage of nitrate that is completely denitrified to dinitrogen gas increases, and the nitrite accumulation in wastewater is suppressed. This observation suggests that the dosed sewer section will remain anoxic for longer periods under lower temperatures <15 °C. Therefore, nitrate dosing is better suited for lower temperatures, for example, from October to April in central Europe. At higher temperatures, the consumption increases, making nitrate dosing costly and ineffective. Therefore, careful consideration to avoid high nitrate dosages for extended periods should be made, especially since the nitrate uptake capability of the biofilm increases with repeated exposure to nitrate (Mohanakrishnan *et al.* 2009).

Given the potential undesirable effects of nitrate dosing, it is worth exploring alternative technologies for sulphide control that can be applied downstream. An alternative to nitrate dosing for downstream sulphide control could be ferrous chloride. The main mechanism here is not oxidation but sulphide precipitation, which was proven to have fast reaction kinetics. However, studies point out that this reaction also needs considerable time (Kiilerich *et al.* 2017), and ferrous will not have an inhibitory effect on methanogens like nitrate (Jiang *et al.* 2013b). Also, there have been trials of dosing pressurised air downstream with a perforated tube around 500 m before the transition to the gravity pipes. With this strategy, good ventilation of the pressure pipe and a positive slide slope of the pipe have to be ensured for a safe operation (Urban 2010; Baxpehler & Urban 2020).

Liquid phase H_2S measurements for monitoring chemical dosing made it possible to closely examine the diurnal sulphide pattern and influence seasonal changes and other flow events. Thereby it was possible to define a profile dosing strategy with a specified time of higher dosages without drastically increasing the overall chemical dosage. However, this dosing profile needs to be constantly updated and checked for changes regarding shifts in seasonal and daily residence time and sulphide profiles. Therefore, installing online H_2S liquid phase sensors such as the SulfiLogger™ is a valuable tool for continuously optimising and fine-tuning the chemical dosing and ensuring that an effective dose-response relationship is achieved. Integrating these sensors in an online dosing control system can regulate the delivery of appropriate chemical dosages according to the measured H_2S, offering an opportunity for water utilities to reduce chemical addition for H_2S control. Given that the SulfiLogger™ only measures unionised sulphide, H_2S, a conversion to total dissolved sulphide is required for characterising the SRB activity (sulphide production rate/ sulphate reduction rate) in the sewer section being dosed. Therefore installing a corresponding pH measurement at the dosing point that is made continuously will improve the accuracy when estimating the sulphide production rates and ultimately improve the optimisation of the control strategy presented in this study.

4. CONCLUSION

The limited downstream nitrate dosing applied performed exceptionally well given the low nitrate concentrations used. The sulphide loads in the upstream wet-well significantly influenced the performance of the downstream nitrate dosing. When the upstream sulphide concentration was less than 0.5 mg L^{-1}, the H_2S measured at the end of the pipe was <0.2 mg L^{-1}. Besides the upstream sulphide concentrations, the residence time of the wastewater slugs arriving at the dosing point and the time these slugs stay in the dosed section were identified as the main reasons for the sulphide spikes frequently occurring between 4:00 and 8:00 a.m. When residence time in the dosed section is >3 hours, sulphide regeneration was likely to occur. The wastewater slugs arriving at the DS between 9:30 p.m. and 03:30 coincide with residences that were >3 hours. The slugs arriving between 4:00 and 8:00 a.m. had the highest residence times, which also contributed to sulphide peaks. To remove the sulphide peaks usually found between 04:00 and 08:00 hours, we recommend increasing the nitrate dosing concentrations for the slugs arriving between 9:30 p.m. and 7:00 a.m. at the DS. This study has demonstrated how continuous monitoring of liquid phase sulphide can be used for optimising chemical dosing. Furthermore, the advantages of downstream nitrate dosing compared to upstream dosing, including chemical savings and odour relief, were clearly shown.

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DATA AVAILABILITY STATEMENT

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

CONFLICT OF INTEREST

The authors declare there is no conflict.

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