Occurrence of 1,4-dioxane in domestic wastewater and processed water: A case study of Thailand's central plain

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Abstract. 1,4-Dioxane is an emerging water contaminant and probable human carcinogen. Its use in various applications and improper waste management can lead to water contamination. This study surveyed 1,4-dioxane contamination in domestic wastewater and water from Thailand's central plain. Samples of influent, effluent, and activated sludge from seven domestic wastewater treatment plants, as well as tap water from 28 public water systems, were collected and analyzed for 1,4-dioxane. The results showed 1,4-dioxane presence in all wastewater samples, with concentrations ranging from 4.34 to 1285.44 μg/L and effluent loads between 0.54 and 254.95 kg/day. Additionally, 1,4-dioxane was detected in five public water systems (17.86% of the sites) at concentrations between 44.54 and 81.24 μg/L. These findings indicate that some municipal water and wastewater treatment plants in Thailand's central plain are not effectively removing 1,4-dioxane, highlighting the need for monitoring and improved treatment technologies to manage contamination in the area.

1 Introduction

1,4-Dioxane ($C_4H_8O_2$), a heterocyclic ether, is a synthetic organic compound utilized in various applications. Primarily, it serves as a stabilizer for chlorinated compounds like trichloroethane. Additionally, it is directly used as a solvent in industries such as textiles, membranes, pharmaceuticals, and paper [1]. Additionally, 1,4-dioxane is a by-product found in numerous consumer products and goods, including shampoo, soap, deodorant, inkjet ink, dyes, degreasers, and in the production of polyethylene terephthalate (PET) plastic [1]. The widespread use and improper disposal of 1,4-dioxane-containing waste can lead to environmental contamination, particularly in aquatic environments.

Due to its unique chemical properties, once 1,4-dioxane contaminates the environment, it becomes pervasive and difficult to remove. The oxygen atoms in the 1,4-dioxane molecule can form hydrogen bonds with water, making it highly soluble and capable of extensive movement from the source zone along with water flow. Furthermore, its stability in the environment and resistance to both biological and chemical degradation are due to its symmetrical structure and strong chemical bonds. With a low octanol-water partition coefficient (log K_{OC} = -0.27), 1,4-dioxane is poorly adsorbed and weakly retained by soil particles. Additionally, its low Henry's law constant (4.88×10^{-6}) suggests that volatilization from water is unlikely [2]. Given these properties, 1,4-dioxane is a persistent organic pollutant with the potential for long-range transport in water environments, including groundwater.

1,4-Dioxane has been identified as a causative agent for liver, peritoneal, and nasal cavity tumors in numerous animal studies. Additionally, the United States Environmental Protection Agency (USEPA) has categorized 1,4-dioxane as a probable human carcinogen (Group B2) [3] and defined that 0.35 μg/L of 1,4-dioxane in water corresponds to a one in a million cancer risk level, making exposure to 1,4-dioxane in drinking water a significant public health concern [4]. Exposure to 1,4-dioxane in drinking water is; thus, a public health concern. Also, the World Health Organization set a guideline value of 50 μg/L for 1,4-dioxane to mitigate its adverse health effects in drinking water [5]. Recently, the USEPA has listed 1,4-dioxane as the first synthetic compound in the Third Unregulated Contaminant Monitoring Rule (UMCR 3), which mandates the analysis of 1,4-dioxane using USEPA Method 522 in all public water systems, with a minimum reporting level (MRL) of 0.07 μg/L [6]. 1,4-Dioxane is frequently found in municipal wastewater, surface water, and drinking water. In Japan, concentrations reached up to 90 mg/L in sewage and 94.8 μg/L in surface water [7]. In Germany, levels of 1,4-dioxane in domestic sewage influent ranged from 262 to 834 ng/L [8]. Wastewater treatment plants were shown to be ineffective at removing 1,4-dioxane, resulting in effluent concentrations between 267 and 62,260 ng/L [8]. This contaminant has also been reported in surface water in the Netherlands, Germany, and the United Kingdom , suggesting human activities as the primary source of contamination [9, 10]. 1,4-Dioxane was also found in drinking and processed water. In California, 1,4-dioxane was detected in 79 drinking water sources and 35 systems

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at concentrations of 1 μg/L or higher [11]. This contamination has led to the closure of drinking water wells in several reports [12, 13]. Furthermore, 1,4-dioxane has been found in groundwater and landfill leachates [9, 12, 14-16].

In Thailand, there is limited information on 1,4-dioxane contamination in water environments. This gap hinders comprehensive ecological and health risk assessments, particularly regarding exposure risk. This study is the first in Thailand to investigate the presence of 1,4-dioxane in wastewater treatment plants and processed water. Additionally, the effectiveness of water and wastewater treatment facilities was evaluated.

2 Materials and Methods

2.1 Chemicals

ACS grade 1,4-dioxane ($C_4H_8O_2$), methylene chloride, sodium sulfate, and hydrochloric acid were obtained from Sigma-Aldrich (Saint Louis, MO). SPEX CertiPrep™ 1,4-Dioxane-d8 and 1,4-dichlorobenzene-d4 were sourced from Thermo Fisher Scientific (Waltham, MA).

2.2 Sample collection

2.2.1 Domestic Wastewater

Influent, effluent, and activated sludge samples were collected from seven domestic wastewater treatment plants operated by the Bangkok Metropolitan Administration (plants A through G; see Figure 1) during 2015-2016. These facilities utilize different treatment technologies for municipal wastewater, as detailed in Table 1. Grab samples were taken in triplicate from each type and location, using 3.8 L sterile polyethylene containers with screw caps. To preserve the wastewater samples, hydrochloric acid was added to adjust the pH to below 2. The samples were then placed on ice and delivered to the laboratory on the same day for 1,4-dioxane analysis.

2.2.2 Processed Water

Water samples from 28 local public water systems in Singburi, Ang Thong, and Phranakhon Si Ayuttaya provinces were collected. These provinces are situated within the Chao Phraya River Basin in Thailand's central plain. Triplicate samples of processed water were gathered in 3.8 L sterile polyethylene containers with screw caps. The samples were acidified with hydrochloric acid to achieve a pH of less than 2, kept on ice, and delivered to the laboratory on the same day for 1,4-dioxane analysis.

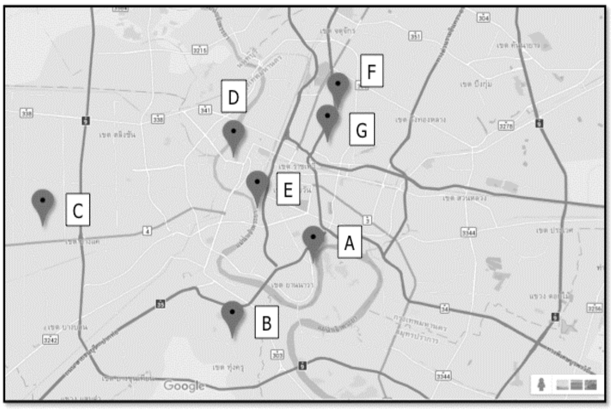

Figure 1. Wastewater Sampling Location in the Bangkok Metropolitan Area, Thailand

Table 1. Wastewater technology used at the studied wastewater treatment facilities in the Bangkok Metropolitan area

Wastewater Treatment Plant	Wastewater Treatment Technology
A	Cyclic Activated Sludge System
B	Vertical Loop Reactor Activated Sludge System
C	Vertical Loop Reactor Activated Sludge System
D	Two-Stage Activated Sludge System
E	Contact Stabilization Activated Sludge System
F	Cyclic Activated Sludge System
G	Activated Sludge with Nutrient Removal

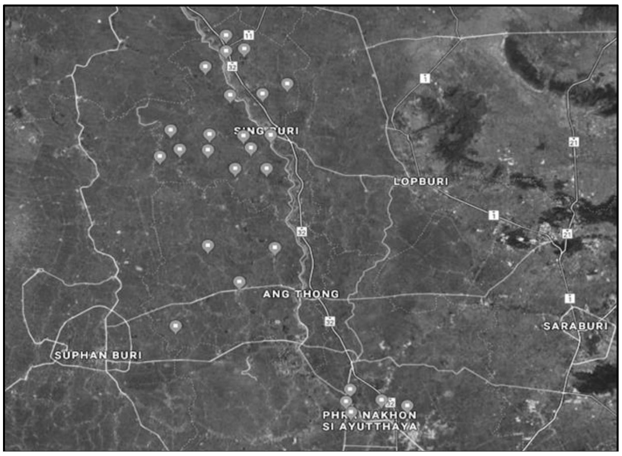

Figure 2. Processed water sampling locations in Singburi, Angthong, and Phranakhon Si Ayuttaya Provinces

2.3 Analytical Method

To quantify 1,4-dioxane concentrations in the ppb (μg/L) range, 200 μL aliquots of the samples were collected and placed in screw-top vials. These samples were preserved and then extracted using a frozen-microextraction method as outlined by Li et al. [17]. Briefly, 200 μL of methylene chloride was added to each sample, along with 200 μg/L of 1,4-dioxane-d8 internal standard and 200 μg/L of 1,4-dichlorobenzene surrogate. After mixing, the vials were placed in a vial tray at -20 ˚C and at a 45˚ angle overnight. The methylene chloride extracts were then collected and transferred to clean vials with inserts. To remove residual water from the extract, 0.05 g of sodium sulfate was added to each vial. A 1 µL aliquot of the extracted sample was injected into an Agilent 7890B Gas Chromatograph (GC) coupled with a 7000C GC/MS Triple Quad mass spectrometry detector (MSD) (Agilent Technologies, Westlake Village, CA) and an HP-5ms column (30 m x 0.25 mm id x 0.25 µm; Hewlett-Packard, Atlanta, GA) using a 7633 ALS autosampler (Agilent Technologies, Westlake Village, CA) in pulse splitless mode. Details of the GC-MSD parameters and methods are provided in Table 2, and the monitored ions for quantification are listed in Table 3.

3 Results and Discussion

3.1 1,4-Dioxane in domestic wastewater

In this study, we examined the presence of 1,4-dioxane in the influent and effluent of seven wastewater treatment plants within the Bangkok metropolitan area. Figure 3 displays the results from analyzing 1,4-dioxane in wastewater samples collected between 2015 and 2016. The findings indicate that all domestic wastewater samples contained 1,4-dioxane. Specifically, the concentration of 1,4-dioxane in influent samples ranged from 122-1183 µg/L in October 2015, 25-449 µg/L in January 2016, and 4-69 µg/L in April 2016. This suggests that human activities are a significant source of 1,4-dioxane contamination in municipal wastewater.

Municipal wastewater contamination with 1,4-dioxane can occur due to the widespread use and disposal of personal care and household products containing this compound. The presence of 1,4-dioxane in these products often stems from the ethoxylation and sulfation processes used in manufacturing various cosmeceutical ingredients, such as polyethylene glycol, polyethylene surfactants, and polyoxyethylene [1]. Recently, 1,4-dioxane has been found in various personal care products, including baby lotion, shampoo, mouthwash, shower gel, body lotion, and toothpaste [1, 18]. Black et al. [19] reported 1,4-dioxane concentrations in ethoxylated raw materials and finished cosmetics as high as 1,410 mg/L and 279 mg/L, respectively. Notably, high levels of 1,4-dioxane, up to 85 ppm, have also been detected in baby shampoo. In Taiwan, 22% of daily-use cosmetics were found to contain 1,4-dioxane at concentrations ranging from 4.2 to 41.1 mg/L [20]. Additionally, 1,4-dioxane has been detected in raw wastewater from the City of Ann Arbor, Michigan, and Kanagawa Prefecture, Japan [7, 15]. To mitigate exposure risks from 1,4-dioxane in personal care products, the Association of Southeast Asian Nations (ASEAN) has established guidelines limiting 1,4-dioxane to 25 mg/kg in cosmetics by 2020 and 10 mg/L by 2023 [21]. These measures are expected to help reduce 1,4-dioxane levels in sewage. While personal care products are likely contributors to 1,4-dioxane in domestic wastewater, further investigation is needed to confirm their role in raw wastewater contamination.

Figure 3 shows that 1,4-dioxane levels in sewage samples varied both spatially and temporally. The average concentrations of 1,4-dioxane in influent samples were 878 µg/L in October 2015, 145 µg/L in January 2016, and 41.6 µg/L in April 2016. Given that 1,4-dioxane is highly soluble in water, it tends to disperse easily under high runoff conditions. Figure 4 illustrates the correlation between rainfall amounts and average 1,4-dioxane concentrations in influent samples from the seven wastewater treatment plants. There is a strong correlation between the amount of rainfall and 1,4-dioxane concentrations, with a high correlation coefficient (R^2 = 0.998). This suggests that rainfall significantly influences the levels of 1,4-dioxane in sewage, consistent with its high solubility and dispersion in runoff conditions.

Table 2. GC- MSD parameters used for the quantitative determination of 1,4-dioxane in municipal water and wastewater samples

Instrument Description	
Instrument:	Triple Quadrupole GC/MS (GC-QQQ) (Agilent Technologies)
GC Model:	Agilent 7890B GC system
MSD Model:	Agilent 7000C GC/MS Triple Quad
GC Column:	HP-5ms part no. 19091S-433UI (30 m × 0.25 mm, 0.25 µm)
Autosampler:	Agilent 7633 ALS
Library:	NIST2011
GC Condition	
Inlet:	Pulse Splitless (1 µL sample) Heater 200 °C Inlet Pressure 10 psi Pulse Pressure 40 psi (2 min) Purge flow 40.3 mL/min (1 min) Carrier gas (Helium) flow 1.3 mL/min
Oven program:	35 °C for 4 min, and then run with a 10 °C/min ramp to 100 °C, followed by a 50 °C/min ramp to 275 °C and held at 275 °C for 3 min
MS Detector:	Solvent Delay 2.8 min m/z Scan range 29-200 Transfer line Temp (Aux Heater) 270 °C EI Source Temp 230 °C, EI Voltage 70 eV

Table 3. Retention times and selection ions for GC- MSD determination

Compound	Molecular ions (m/z)	Retention time (min)
1,4-dioxane	43, 58, 88	3.007
1,4-dioxane-d8	46, 64, 96	3.061
1,4-dichlorobenzene-d4	115, 150, 152	10.228

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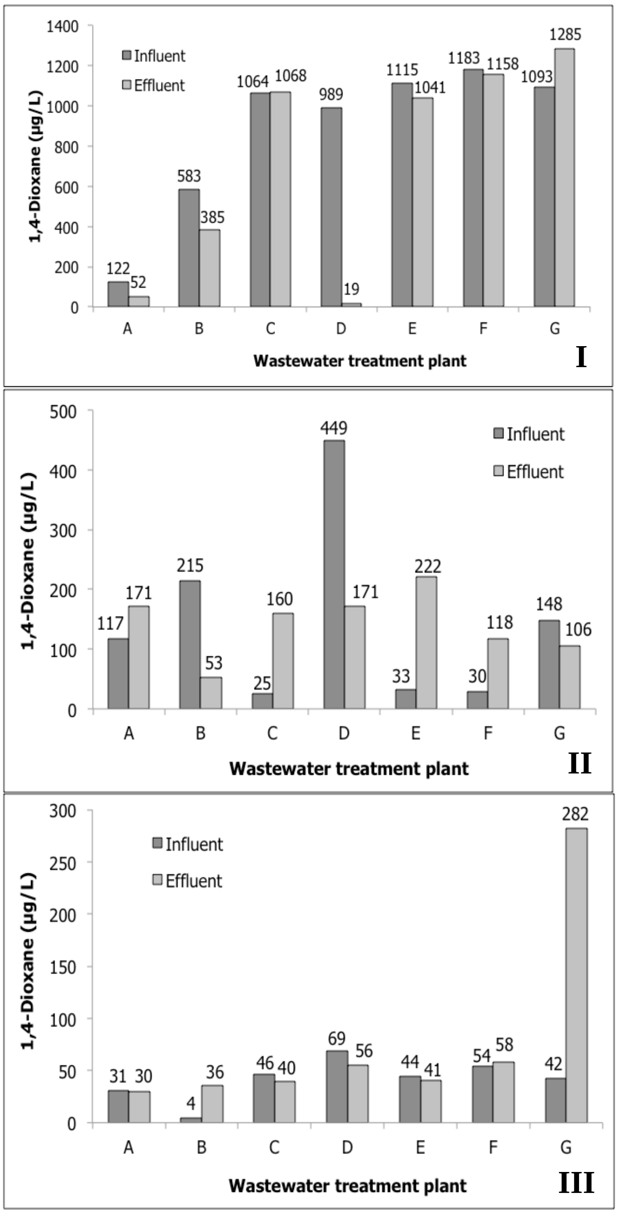

Figure 3. 1,4-Dioxane concentration in influent and effluent of wastewater treatment plants in Bangkok Metropolitan Area, (I) data of October 2015, (II) data of January 2016, (III) data of April 2016.

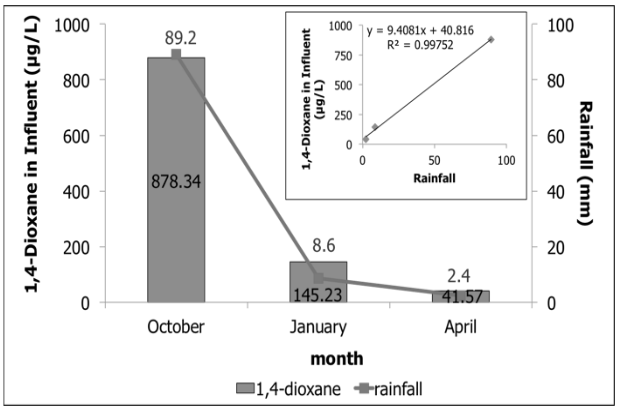

Figure 4. Relationship between amount of rainfall and average 1,4-dioxane concentration in the influent of wastewater treatment plants. The concentration of 1,4-dioxane in the influent directly varied as the amount of rainfall

3.2 Treatment of 1,4-dioxane by wastewater treatment plants

To assess the performance of wastewater treatment plants in removing 1,4-dioxane, influent and effluent samples from seven plants were tested three times over a six-month period, encompassing both rainy and dry seasons. Figure 3 presents a comparison of 1,4-dioxane concentrations in influent and effluent samples. During the rainy season (October 2015), plants A, B, and D achieved significant reductions in 1,4-dioxane, with removal efficiencies of 57.4% (from 122 to 52.0 µg/L), 34.0% (from 583 to 385 µg/L), and 98.1% (from 989 to 19.0 µg/L), respectively. In winter (January 2016), plants B, D, and F demonstrated removal rates of 75.3% (from 215 to 53.1 µg/L), 61.9% (from 449 to 171 µg/L), and 28.4% (from 148 to 106 µg/L), respectively. However, in summer (April 2016), only plant D managed to treat 1,4-dioxane with an efficiency of 18%. Among the facilities studied, plant D consistently showed the highest effectiveness in removing 1,4-dioxane across all seasons. Plant D utilizes a Two-Stage Activated Sludge system with sequential aeration and clarifier tanks for treating municipal wastewater (Figure 5). Despite this, the concentrations of 1,4-dioxane in the influent of nearly all plants were similar to those in the effluent, indicating that these treatment processes are not optimized for 1,4-dioxane removal.

Microorganisms in wastewater treatment plants are crucial for the biodegradation of 1,4-dioxane. Laboratory studies have identified several bacterial and fungal strains that can effectively degrade 1,4-dioxane through catalytic processes [22, 23]. Under aerobic conditions, various monooxygenase enzymes have been shown to facilitate the biodegradation of 1,4-dioxane [24, 25]. This degradation can occur via metabolic or co-metabolic pathways. In the metabolic pathway, 1,4-dioxane serves as the sole carbon and energy source for microbial growth [22, 23]. Conversely, co-metabolism requires the presence of primary substrates like tetrahydrofuran, methane, or propane to stimulate the production of enzymes that aid in the degradation of 1,4-dioxane [24, 26]. Anaerobic conditions also support 1,4-dioxane degradation; for instance, Shen et al. [27] found that iron-reducing bacteria in anaerobic sludge could degrade up to 40% of 1,4-dioxane without any chemical additions. The reduction in 1,4-dioxane concentration in wastewater suggests that the activated sludge from wastewater treatment plants A, B, D, and G may harbor microorganisms capable of degrading 1,4-dioxane. However, the effectiveness of these microorganisms can be constrained by factors such as the presence of metals and organic solvents [28, 29]. Additionally, insufficient levels of 1,4-dioxane may not support the growth of microorganisms responsible for metabolic biodegradation, leading to a diminished capacity for 1,4-dioxane removal in certain treatment plants. Consequently, the use of biological treatment systems for addressing 1,4-dioxane in domestic wastewater and groundwater remains constrained.

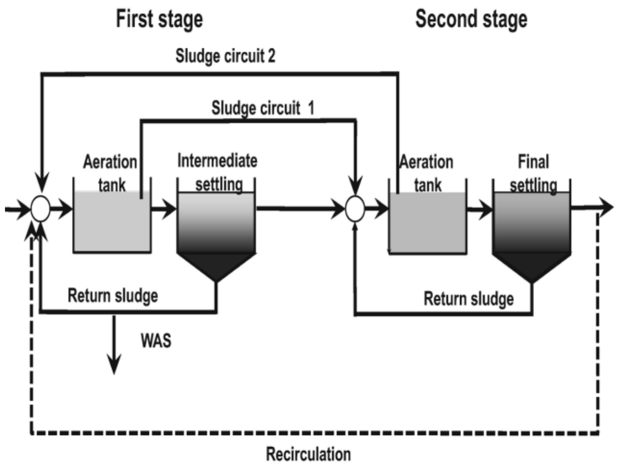

Figure 5. Schematic diagram of Two-Stage Activated Sludge system

In several instances, the concentration of 1,4-dioxane in effluents was found to be higher than in influents [8]. The elevated 1,4-dioxane levels observed in the effluent of wastewater treatment plant G may be attributed to the use of methanol containing 1,4-dioxane as an impurity during the denitrification process (a nutrient removal step). This observation aligns with the findings of Stepien et al. [8] , who reported high 1,4-dioxane concentrations in the effluent of a sewage treatment plant employing a post-anoxic denitrification process, where methanol with 1,650 µg 1,4-dioxane/mL was utilized. Additionally, some evaporation of water could have occurred, resulting in a higher concentration of the non-volatile compound within the treatment facilities.

3.3 Load of 1,4-dioxane from wastewater treatment plants

It was evident that 1,4-dioxane is a contaminant found in the effluent of municipal wastewater treatment plants in Bangkok. Table 4 displays the 1,4-dioxane load discharged by the seven wastewater treatment plants, with Wastewater Treatment Plant G having the highest load at 449.8 kg/d, and Wastewater Treatment Plant D having the lowest at 2.2 kg/d. The presence of 1,4-dioxane in the effluent samples indicates that these plants could be contributing to 1,4-dioxane contamination in the aquatic environment. However, the 1,4-dioxane loads varied over time, being highest during the rainy season and lowest in the summer. This suggests that there are significant temporal fluctuations in the 1,4-dioxane load in the Chao Phraya River, where the effluents are discharged. Consequently, random sampling and our findings may not fully capture the true extent of 1,4-dioxane transport in the river, leading to potential underestimation or overestimation.

Table 4. 1,4-dioxane load from various wastewater treatment plants in Bangkok

Wastewater Treatment Plant	Effluent Volume (m^3/day)	1,4-Dioxane Concentration (µg/L)	Release (kg/day)
A	200,000	30 – 171	6.0 – 34.2
B	65,000	36 – 385	2.3 – 25.0
C	157,000	40 – 1,068	6.2 – 167.7
D	40,000	56 – 171	2.2 – 6.84
E	30,000	41 – 1,041	1.2 – 31.23
F	150,000	58 – 1,158	8.7 – 173.7
G	350,000	106 – 1,285	37.1 – 449.8

3.4 1,4-Dioxane in the processed water

1,4-Dioxane has contaminated many drinking water sources. Its resistance to natural biodegradation allows it to persist in water systems, potentially reaching dangerous levels. Classified as a probable human carcinogen by the U.S. Environmental Protection Agency, its presence in drinking water poses significant health risks [3]. As a result, municipalities and environmental agencies are focusing on monitoring 1,4-dioxane concentrations and investigating advanced treatment methods, such as advanced oxidation processes, to effectively remove it from water sources [1, 6]. Maintaining safe levels of this contaminant is crucial for public health, and ongoing evaluation is essential for understanding and managing its risk.

In this study, we assessed the presence of 1,4-dioxane in 28 processed water samples from three provinces: Pranakornsriayuttaya, Singburi, and Angthong, all located along the Chaophraya River in Thailand's central plain. Our analysis revealed that 1,4-dioxane was detected in five of these samples, approximately 17.86% of the total. The concentrations in these samples ranged from 44.58 to 81.24 µg/L (Figure 6). Notably, samples pp25, pp26, and pp28 exceeded the drinking water standards established by the World Health Organization [5]. All contaminated samples were from Pranakornsriayuttaya province, where groundwater is used as the source for processed water. This suggests that groundwater in Pranakornsriayuttaya may be contaminated with 1,4-dioxane, making the water unsafe for drinking. Therefore, it is crucial to implement treatment solutions for 1,4-dioxane in groundwater or use additional point-of-use treatments to safeguard public health.

1,4-Dioxane is prevalent in groundwater due to its high solubility and resistance to biodegradation, leading to growing global concern about its contamination. This issue has been reported in various regions, including the United States, Canada, Europe, Asia, and Australia [1, 30], emphasizing its worldwide significance. Once 1,4-dioxane enters groundwater, it spreads rapidly, potentially affecting large areas. Identifying the source of this contamination is critical. The primary sources of 1,4-dioxane contamination are its historical use as a stabilizer in industrial solvents and its presence in various consumer products, including cosmetics,

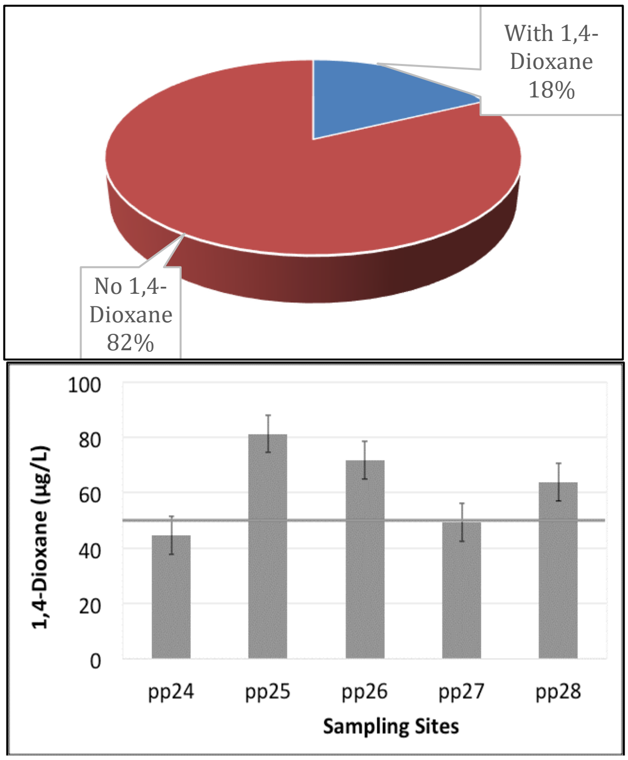

Figure 6. 1,4-Dioxane concentration in processed water collected from five locations where 1,4-dioxane was detected. Three of these exceeded the World Health Organization's guideline value of 50 µg 1,4-dioxane/L (line).

pharmaceuticals, and household cleaners [1]. Additionally, landfill leachate may also contribute to 1,4-dioxane contamination. To address this, further investigation is needed to pinpoint the source of 1,4-dioxane contamination in Pranakornsriayuttaya. Monitoring wells should be used to track 1,4-dioxane levels and understand the direction and extent of contamination plumes. This data is crucial for public health protection and effective pollution management.

Removing 1,4-dioxane from groundwater poses significant challenges. While Advanced Oxidation Processes (AOPs) are among the most effective and commonly employed techniques for large-scale treatment of 1,4-dioxane-contaminated water [1, 31], it is crucial to monitor the formation of byproducts and the potential increase in disinfection byproducts (DBPs) [32, 33]. Although traditional and modified adsorbent materials can temporarily remove 1,4-dioxane, they require additional disposal and regeneration steps [34, 35]. Bioreactors [36-38], biofiltration systems [39, 40], and biosparging systems [41-43] have shown promise for biodegrading 1,4-dioxane, but meeting stringent drinking water standards cost-effectively remains a challenge. To achieve effective biodegradation and comply with drinking water guidelines, it may be necessary to identify limiting growth factors and pre-treat chlorinated solvents [44, 45]. Combining physical-chemical methods with biodegradation may offer a synergistic solution for effective 1,4-dioxane removal and cost efficiency [46-49].

4 Conclusion

This study reveals a critical issue: 1,4-dioxane is prevalent in the water environments of Thailand's central plain, affecting both municipal wastewater and processed water. The extensive detection of 1,4-dioxane in these water sources, combined with its low removal efficiency in current treatment processes and potential health risks, underscores an urgent need for action. Establishing stringent water quality standards for 1,4-dioxane is not just advisable but essential to safeguard public health and enhance water quality in Thailand. Addressing this issue proactively will ensure cleaner, safer water for all.

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