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Removal of Triclosan at A Full-Scale Wastewater Treatment Plant in Baltimore

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Abstract: Triclosan is a persistent antimicrobial contaminant frequently detected in municipal wastewater effluents due to its incomplete removal during conventional wastewater treatment processes. This study evaluates the seasonal concentration and removal of triclosan across two full-scale WWTPs relative to their fluctuating physicochemical properties. Grab samples of primary influent (PI), secondary effluent (SE), and tertiary effluent (TE) were collected across four seasons and quantified using solid-phase extraction and spectrophotometric analysis to identify potential drivers of treatment variability. The results demonstrated marked seasonal variation in triclosan concentrations, with the highest influent concentrations detected during summer, while the lowest concentrations were found during spring for Plant A and winter for Plant B. Triclosan removal efficiency also varied seasonally, ranging from approximately 20% in winter to 75% in summer. While primary and secondary processes achieved moderate removal via hydrophobic partitioning, a systemic decline in tertiary removal efficiency was observed during winter. Pooled Pearson correlation analysis revealed significant positive correlation between temperature and removal efficiency, indicating thermal dependency while the inverse relationship between removal efficiency and salinity ($r = -0.68$, $p = 0.063$) suggests a potential inhibitory trend on triclosan mitigation. Based on quantifiable triclosan concentrations above the limit of quantification (LOQ; 2.66 µg/L), tertiary effluent concentrations (<2.66–4.72 µg/L) exceeded the adopted PNEC of 0.05 µg/L, suggesting considerable ecological risk, with risk quotient (RQ) values peaking at 94.4. Although plant B exhibited superior triclosan removal efficiency, quantifiable residual triclosan persisted in the tertiary effluents of both facilities. Overall, the findings highlight the importance of seasonal monitoring and optimization of tertiary treatment processes to improve triclosan removal in municipal wastewater treatment plants. However, the analytical LOQ precluded assessment of ecological risks at lower triclosan concentrations, warranting future monitoring using more sensitive analytical method. The results further demonstrate that urban WWTPs may act as sources of pseudo-persistent triclosan discharge, emphasizing the need for advanced matrix-adaptive treatment technologies and enhanced source-control strategies to mitigate the environmental risks associated with persistent emerging contaminants.

Keywords: triclosan; WWTP effluents; municipal wastewater; wastewater physicochemistry; micropollutant removal; tertiary wastewater treatment; risk quotient; pseudo-persistence

1. Introduction

Water pollution is a serious environmental and public health concern in America as a result of myriads of both natural and anthropogenic activities taking place on the continent [1]. The level of pollution is so high that the Environmental Protection Agency (EPA) have identified about 70,000 water bodies nationwide that fail to meet water quality standards [2]. A large number of organic pollutants (triclosan, phenols, polychlorinated biphenyls, etc.), inorganic pollutants (Cd, Pb, Hg, Cr, and Cu), as well as microbes, have been found in polluted water bodies [1,3]. Many of these pollutants are potentially toxic and are capable of causing kidney damage, cancer, nervous system damage, gastrointestinal damage, and circulatory system issues [1]. Triclosan (5-chloro-2-(2,4-dichlorophenoxy)phenol) (Figure 1) is a synthetic aromatic compound, commercially referred to as Irgacare MP or Irgasan DP 300 [4].

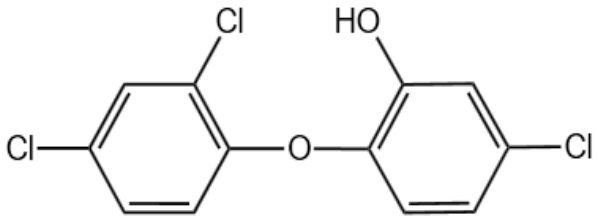

Figure 1. Chemical Structure of Triclosan. Adapted from Ref. [5] under a Creative Commons Attribution (CC-BY) license.

It is widely utilized as a broad-spectrum antimicrobial agent in a variety of pharmaceutical and personal care products (PPCPs) such as hand sanitizer, soap, deodorant, skin cream, toothpaste, detergents, clothing textiles, furniture, and cosmetics [6,7]. It is also widely utilized clinically as an antiseptic in surgical sutures, implants, scrubs, and medical devices [8]. The annual production of triclosan was estimated to be about 1500 tons, of which 132 million liters are reported to be consumed in the United States [6]. Triclosan is a highly lipophilic compound capable of dermal absorption through the skin or by oral absorption through the gastrointestinal tract (GIT) [9]. As a result, it has been detected in a range of organisms including algae, earthworms, and fish, as well as in various human body fluids such as urine, blood, and breast milk [6]. In a study conducted between 2003 and 2004, triclosan was detected in the urine in 75% of the US population at a concentration ranging from 7.9 nM to 13.1 nM [8]. Humans are continually exposed to triclosan through the use of PPCPs containing Triclosan for personal grooming, or through consumption or ingestion of triclosan-contaminated food and water [9,10] (Figure 2).

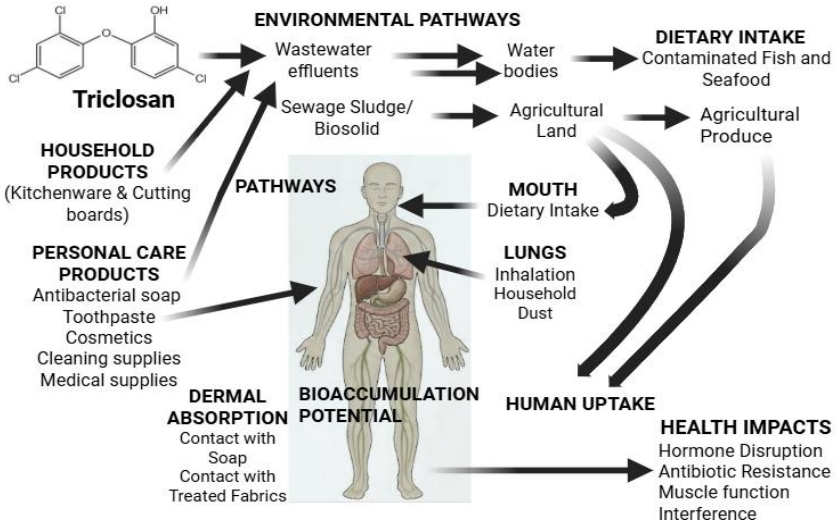

Figure 2. Sources and Pathways of Human Exposure to Triclosan. Adapted from Ref. [10], with illustrative and conceptual modifications based on findings from Ref. [5,9,11].

Triclosan has also been detected in soils and in various water bodies around the world, including rivers, lakes, drinking water, estuarine, and coastal waters [4]. Their presence has been linked to the discharge of industrial, agricultural, and personal care products containing triclosan into drains and sewers, ultimately entering the wastewater system [9].

In addition to its detection in wastewater influents, the persistence, hydrophobicity, and partial resistance of triclosan to biodegradation have also led to its presence in wastewater effluents, sewage sludge, and the receiving aquatic environments [9,12]. In North America, it has been detected at between 75 to 100% of rivers, lakes and estuarine waters at a concentration ranging between 1–34.9 ng/L, in Asia it was found at concentration ranging from 18–1023 ng/L, in Europe it was detected at concentration between 0.2 to 285 ng/L, while in Oceania it was detected at concentration between 3 to 75 ng/L [12]. A study conducted on 139 streams in 20 US states between 1999 and 2000, also showed the presence of triclosan in over 57% of the streams sampled at a concentration up to 2300 ng/L [13].

Triclosan is a hazardous substance that has been found to cause endocrine disruption in rats, sheep, fish, and amphibians [5,12]. It is also harmful to the liver, promotes the development of liver tumors, induces bacterial resistance to other antimicrobial chemicals, exacerbates allergies and skin sensitivities, and promotes cancer development [9]. Triclosan also causes spontaneous abortion and developmental problems in humans [14], and is harmful to aquatic species, algae, and some invertebrates [15]. The degradation products of triclosan, such as dioxin, and the chlorinated derivatives, including 2,4-dichlorophenol and 2,4,6-trichlorophenol, are also highly toxic [16]. Although conventional wastewater treatment plants (WWTPs) can partially remove triclosan through sorption and biodegradation processes, the heterogeneity of wastewater matrices [17], and the inherent limitations of conventional treatment technologies frequently result in incomplete removal and the persistence of environmentally relevant triclosan concentrations and other micropollutants in treated effluents [18,19]. This has resulted in the detection of triclosan in treated wastewater (effluents) at concentrations ranging from 10 ng/L to 2210 ng/L [12]. A considerable amount of this unremoved triclosan eventually makes its way into lakes, rivers, soils, sediments, and swimming pools [20]. Furthermore, triclosan can also get into these water bodies through sewage overflows, agricultural runoff from applied fungicide and/ or bactericides or from triclosan adsorbed onto biosolids applied to the farms [12]. The fact that rivers and lakes serve as a major source of water for irrigation and drinking water for humans and animals, makes the tracking of triclosan content in wastewater influents and effluents a matter of urgency due to their toxic and potentially toxic effects on humans, aquatic organisms, and the environment. Recent advances in wastewater treatment research have demonstrated the increasing effectiveness of advanced and hybrid technologies for the removal of pharmaceuticals and personal care products (PPCPs), including triclosan [21]. Advanced technologies-ranging from physical separation via membranes and adsorption to destructive radical-driven and electrochemical oxidation have shown considerable potential for enhancing micropollutant degradation and mineralization [22,23]. Furthermore, hybrid treatment systems integrating biological and advanced tertiary processes are increasingly being explored due to their improved performance in removing persistent emerging contaminants from municipal wastewater effluents [21]. Despite advancements in technology, the efficiency of triclosan removal varies significantly among wastewater treatment plants (WWTPs). This variability may be influenced by factors such as operational conditions, the physicochemical properties of wastewater, seasonal fluctuations, and the configuration of treatment systems. While there is extensive documentation regarding the presence of triclosan in WWTPs, few studies have combined seasonal monitoring, physicochemical characterization, and ecological risk assessment in large-scale operational tertiary treatment systems. Consequently, there is a critical need for site-specific investigations evaluating triclosan occurrence and removal performance under real environmental conditions. This study therefore investigated the seasonal variation and removal efficiency of triclosan across primary, secondary, and tertiary treatment stages in two municipal WWTPs, while also assessing the influence of wastewater physicochemical characteristics on triclosan distribution and treatment performance. In addition, the applicability of an SPE-coupled spectrophotometric method was evaluated as a cost-effective approach for triclosan quantification in complex wastewater matrices.

2. Materials and Methods

2.1. Study Area

The study focused on two municipal wastewater treatment plants (WWTP-A and WWTP-B) located in Baltimore. Plant A, with an average flow of 63–73 MGD, utilizes oxygen-aerated activated sludge and biological nutrient removal, while plant B, with a 180 MGD capacity, utilizes fine-bubble aeration, sand filtration, and chemical phosphorous removal via the use of waste pickle liquor derived from a steel mill (Table 1).

Table 1. Wastewater Treatment Schematics of the two WWTPs.

Feature	WWTP-A	WWTP-B
Average treatment capacity	~63–73 MGD average flow with peaks ~103 MGD	~180 MGD with >400 MGD
Primary waste streams	Municipal sewage (90%), industrial waste (10%)	Municipal sewage plus industrial wastewaters
Preliminary treatment	Screening + grit removal	Fine screening + grit removal
Primary treatment	Primary sedimentation tanks	Multiple primary sedimentation tanks
Secondary treatment	Activated sludge (pure oxygen aeration)	Activated sludge (fine bubble aeration)
Tertiary/ advanced treatment	Disinfection (chlorination, dechlorination)	Sand filtration + disinfection
Nutrient control process	Biological nutrient removal	Chemical addition (pickle liquor, ferric chloride) + biological nutrient removal
Special additives	None reported for coagulation/precipitation	Waste pickle liquor for phosphorous removal
Sludge handling method	Thickening, dewatering, pelletization, or land application.	Gravity belt thickeners, digesters, further solids processing.
Service Population	~450,000	~1.3 million

Note: Fine-bubble aeration is a type of diffused aeration technology that utilizes ambient air to enhance oxygen transfer efficiency in activated sludge systems. (Source: [24,25]).

2.2. Sample Collection

Wastewater samples were collected seasonally (spring, autumn, winter, and summer), using a grab sampling technique, from the main influent (PI), secondary effluent (SE), and tertiary effluent (TE) of the two treatment plants. All grab samples from primary influent, secondary effluent and tertiary effluent were collected in triplicates between 10:00 am and 11:00 am to capture the system's performance during the morning's peak loading period. The grab samples were collected into pre-rinsed 1L HDPE bottles, acidified to pH 2, and immediately placed in a dark cooler and transported to the labs on ice, in the Autumn, spring, summer and winter to capture seasonal impacts.

2.3. Justification for Grab Sampling

The 24-h composite sampling is a conventional standard for bulk-wastewater parameters, however a discrete grab sampling techniques was strategically utilized in this study to maintain triclosan chemical integrity. Triclosan is a highly hydrophobic compound with a high octanol-water partition coefficient (K_{ow} of 4.8 or greater) at pH 6.7, and exhibits a strong affinity for organic phases and polymeric materials [26,27]. Therefore, prolonged residence during composite sampling may increase the likelihood of triclosan partitioning to suspended solids and contact surfaces, as well as potential physicochemical changes during storage. Previous studies have demonstrated adsorption of triclosan onto plastic materials, suggesting that prolonged contact with polymeric components, such as sampling tubing and storage containers, may introduce potential analyte losses during automated composite sampling [28,29]. Therefore, grab sampling was selected to capture the instantaneous distribution of triclosan between dissolved and particle-associated phases while minimizing potential sampling artifacts associated with extended sample residence and storage. Samples were collected and preserved in accordance with established analytical guidance for pharmaceuticals and personal care products [30].

2.4. In-Situ Physicochemical Analysis

The physicochemical properties (temperature (°C), pH, electrical conductivity (EC, µS/cm), total dissolved solids (TDS, mg/L), and salinity (ppm)) of the wastewater samples were measured in situ after the collection of each grab sample (n = 3), using the Ohaus™ Starter ST20M-C Multi-parameter pen meter (Ohaus Corp., Parsippany, NJ, USA). To ensure data integrity, measurements were conducted within 15 min of sample collection to prevent errors associated with temperature equilibration or gas exchange. The multi-parameter pen was calibrated daily prior to sampling. pH calibration was carried out using a three-point method using standard buffer solutions (pH 4.01, 7.00, and 10.01). Conductivity calibration was performed using a single-point calibration with a 1413 µS/cm standard solution (Ohaus Corp., Parsippany, NJ, USA) at 25 °C according to the manufacturer's instructions. Between each replicate and sampling point (influent, secondary, and tertiary), the electrode was thoroughly rinsed with deionized water to prevent cross-contamination.

2.5. Sample Extraction and Enrichment

The wastewater samples collected (500 mL) were first stabilized by high-speed refrigerated centrifugation (4700 RPM, 30 min, 4 °C) and the resulting supernatant filtered through 0.2 µm syringe filters to eliminate particulate debris. Solid-phase extraction (SPE) was then utilized for sample extraction and enrichment to overcome the complexities of the wastewater matrix and the trace concentrations of the analyte. Waters Oasis HLB SPE cartridges (200 mg, 6 cc) were utilized in accordance with EPA Method 1694 guidelines for extraction of pharmaceuticals and personal care products in wastewater matrices [30]. The SPE cartridges were conditioned with 5 mL of HPLC-grade methanol followed by equilibration with 5 mL of ultrapure water. The sample was then loaded onto the cartridges at a controlled flow rate of 5 mL/min using vacuum manifold equipped with large-volume PTFE sampling tubes and polypropylene cartridge adaptors. The sorbent was washed with 5 mL of 5% aqueous methanol solution to eliminate matrix interference and dried for 15 min under vacuum to eliminate residual moisture. The retained triclosan was then eluted with 5 mL of methanol. This resulted in a 100-fold analyte enrichment and significantly improving triclosan detectability within the complex wastewater matrix.

The eluted analyte was preserved at 4 °C in the dark immediately after collection to inhibit microbial degradation and minimize adsorption. Spectrophotometric evaluation of the triclosan content was initiated within 48 h of extraction as part of the laboratory analytical protocol.

The coupling of SPE-based pre-concentration with spectrophotometric evaluation allowed for the determination of triclosan levels across varying seasonal matrices with high sensitivity, effectively bridging the gap between trace-level occurrence and the detection limits of the instrument.

2.6. Triclosan Evaluation and Validation

The triclosan content of the wastewater was evaluated using a diazotization-coupling reaction (Figure 3) according to the method described by Lu et al. [31] with slight modification. In this method, 5 mL of the eluate was mixed with 5 mL of ice cold 0.004 mol L^{-1} NaOH. Sodium nitrite solution (0.020 mol L^{-1}) was also prepared. In order to prepare the diazonium salt, 1.4 mL of the sodium nitrite solution was transferred to a 10 mL volumetric flask and to this was added 1 mL of 0.25% *p*-sulfanilic acid solution in 0.09 mol L^{-1} hydrochloric acid. The diazonium ion produced was then reacted with 3.5 mL of the eluate-NaOH mixture followed by the addition of 2.2 mL of alkaline glycine buffer solution. Finally, the solution was diluted with 1.9 mL of deionized water.

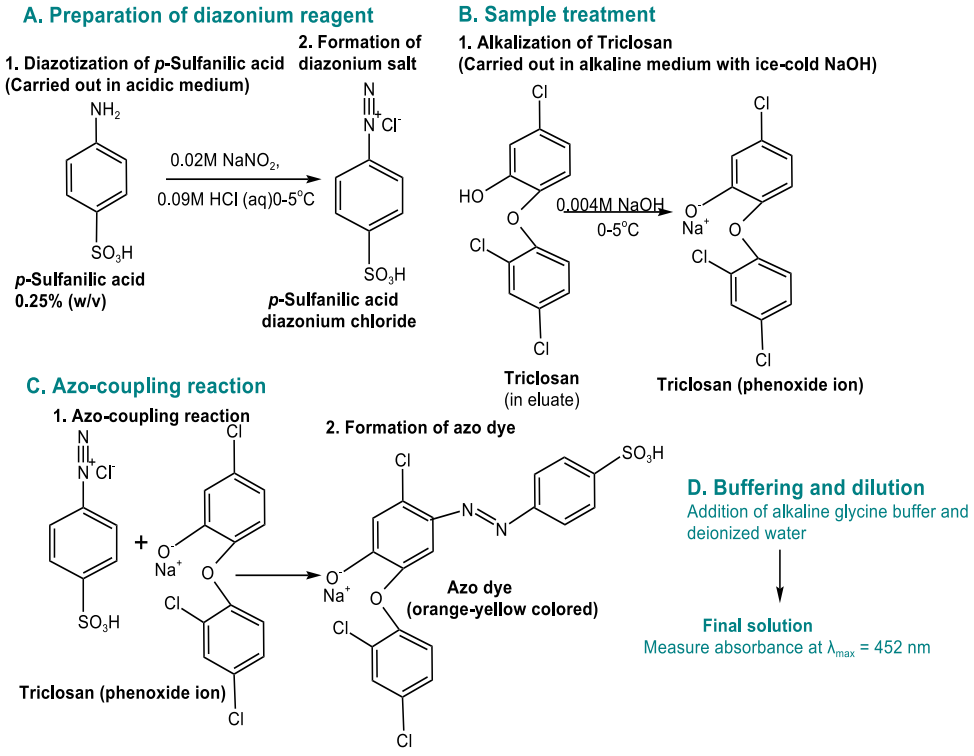

Figure 3. Proposed reaction mechanism for Spectrophotometric determination of triclosan using diazotization reaction. Adapted from Ref. [30], with optimization and pathway concepts based on findings from Ref. [11,32], and experimental conditions modified by the authors from [31].

The alkaline environment provided by the sodium hydroxide solution, aided by the glycine buffer, ensured the formation of reactive phenolate ion of triclosan which then attacks the diazonium ion resulting in the formation of an intense and stable yellow azo-dye. Ice-cold sodium hydroxide solution was used to stabilize the diazo-coupling reaction. The blank reagent solution was performed in the same manner with the NaOH solution without the eluate. The absorbance of the azo dye was measured at the maximum absorption wavelength of 452 nm using a Biospec-1601 UV–vis spectrophotometer, Shimadzu Corporation, Kyoto, Japan. This process was repeated for the PI, SE and TE obtained from the two WWTPs, and all experiments were performed in triplicate. A series of triclosan standard concentrations ranging from 3.125 to 50 μgL^{-1} were utilized to generate a calibration curve (Figure 4). The resulting regression equation ($y = 0.0229x + 0.0166$, $R^2 > 0.9983$) was utilized to determine the triclosan concentration in the environmental samples. Through the utilization of 100-fold SPE enrichment, the method achieved a limit of detection (LOD) of 0.80 µg/L and a limit of quantification of 2.66 µg/L (Table 2). Wastewater samples containing triclosan target responses falling below the LOQ threshold could not be reliably quantified and were reported as < 2.66 µg/L to maintain analytical rigor. Ultimately, the quantified concentrations of triclosan measured across the collected wastewater samples ranged from < 2.66 µg/L to 10.82 µg/L.

The precision of the method was confirmed with a relative standard deviation (RSD) < 5% (n = 3) at the LOQ, thereby demonstrating high reproducibility. The efficiency of the Waters Oasis HLB extraction and the impact of the wastewater matrix was evaluated using spike recovery experiment. Samples of the raw primary influent (PI) and raw tertiary effluent (TE) were spiked with known concentrations of triclosan (4.0 µg/L) prior to SPE extraction. The mean recovery for PI was 85.27 ± 0.4, SE was 87.9 ± 0.42 while the mean recovery for TE was 87.4 ± 0.36. The high recovery observed confirms that the 5% methanol wash effectively reduced matrix interference without premature triclosan elution, while the methanol elution step successfully recovered the adsorbed triclosan from the HLB sorbent. The RSD for the three matrices were below 1% indicating high repeatability.

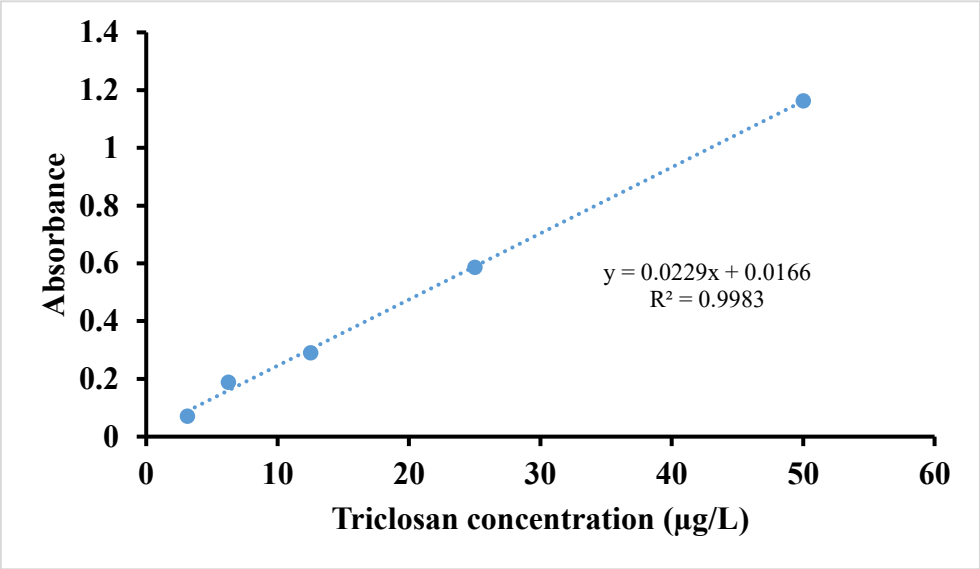

Figure 4. Triclosan Calibration Curve (µg/L).

Table 2. Analytical Performance and Method Validation for Triclosan Determination.

Parameter	Validation Result	Reference/Acceptance Criteria
Linear calibration range	3.125–50 µg/L	Instrument linearity ($R^2 > 0.9983$)
Measured environmental range	<2.66–10.82 µg/L	Quantified concentrations across collected wastewater samples
Limit of detection (LOD)	0.80 µg/L	Calculated as 3x SD of the lowest detected signal
Limit of quantification (LOQ)	2.66 µg/L	Set as the lowest concentration quantified with RSD < 5%.
Enrichment factor	100x	500 mL to 5 mL (According to EPA 1694)
Mean recovery (%)	PI (85.27 ± 0.4), SE (87.9 ± 0.42), TE (87.4 ± 0.36).	Matrix spike (4.0 µg/L)

Note: <2.66 µg/L indicates concentrations below the method's Limit of Quantification.

2.7. Statistical Analysis and Matrix Correlation

All experimental data were processed in triplicate, and results are expressed as mean and standard deviations. The relationship between the wastewater matrix constituents (temperature, TDS, EC, salinity, and pH) and triclosan mitigation (overall removal efficiency (ORE) and tertiary removal efficiency (TRE), was evaluated using pooled Pearson correlation analysis (IBM SPSS Statistics (v.25.0). Statistical significance was defined at $p < 0.05$, with $p < 0.01$ indicating highly significant correlations. While p-values between 0.05 and 0.10 were considered as indicating a strong directional trend. A one-way analysis of variance (ANOVA) was conducted, and means were separated using Tukey's HSD.

ORE was determined by comparing the triclosan concentration in the PI with the concentration in the final TE, is calculated as:

$$ORE\ (\%) = \frac{C_{PI} - C_{TE}}{C_{PI}} \times 100 \quad (1)$$

TRE was determined by comparing the triclosan concentration in the SE (which serves as the influent to the tertiary stage) with the concentration in the final TE, is calculated as:

$$TRE\ (\%) = \frac{C_{SE} - C_{TE}}{C_{SE}} \times 100 \quad (2)$$

where C_{PI}, C_{SE} and C_{TE} represent the primary influent, secondary effluent, and the tertiary effluent concentrations (µg/L), respectively.

The analyte loss during the enrichment process was accounted for by correcting for the recovery. The measured values were divided by the mean recovery factor determined for each specific matrix (0.853 for PI, 0.879 for SE, and 0.874 for TE).

The ecological risk posed by the discharged effluents was determined using the risk quotient (RQ) method.

$$RQ = \frac{MEC}{PNEC} \quad (3)$$

where MEC represents the measured environmental concentration in the TE, while PNEC represents the predicted no-effect concentration for triclosan (0.05 µg/L).

3. Results and Discussions

3.1. Seasonal and Treatment Stage Variation of Triclosan Concentrations

The concentrations of triclosan detected in the primary influent, secondary effluent, and tertiary effluent streams of the two wastewater treatment plants (WWTP-A and WWTP-B) across the four seasons (Table 3) showed that all treatment phases and seasons showed detectable triclosan levels, suggesting that both plants received constant triclosan input over the observation period. This is in agreement with the observations made by [15].

3.2. Treatment Efficiency and Process Performance

In plant A (Table 3), the triclosan concentration in primary influent varied between 5.20 (spring) and 9.33 µg/L (summer), while in tertiary effluent it ranged from 3.1 (summer) to 4.72 µg/L (winter). In plant B (Table 3), influent values varied from 5.5 (winter) to 10.82 µg/L (summer) while tertiary effluent ranged from <2.66 (summer) to 4.40 µg/L (winter). The higher influent triclosan observed in plant B, in all except winter season, may be attributed to its significantly larger residential service population and a dense concentration of urban medical hubs, which serve as primary point sources for complex pharmaceutical influent [33]. The elevated summer triclosan concentration is primarily attributable to increased triclosan discharge due to seasonal increase in the use of triclosan-containing substances, increased industrial activity, coupled with the elevated temperatures promoting desorption of hydrophobic triclosan from sediments and run off. The observed triclosan concentration in this study, though slightly lower than the 3.8–16.6 µg/L observed in PI by [34], is much higher than the 0.32–0.47 µg/L observed in PI in four WWTPs by [35]. It is also higher than the 0.66–0.73 µg/L detected in wastewater by [36].

Table 3. Seasonal Concentrations of Triclosan and their treatment efficiencies in WWTPs A and B.

Plant	Season	PI (µg/L)	SE (µg/L)	TE (µg/L)	TRE (%)	ORE (%)
WWTP-A	Spring	5.20 ± 0.02	4.30 ± 0.03	3.50 ± 0.15	18.6 *	32.69 *
	Autumn	6.50 ± 0.07	5.20 ± 0.04	4.60 ± 0.14	11.54 *	29.23 *
	Winter	6.20 ± 0.26	5.10 ± 0.20	4.72 ± 0.13	7.45 *	23.87 **
	Summer	9.33 ± 0.23	6.72 ± 0.17	3.10 ± 0.06	53.87 *	66.77 **
WWTP-B	Spring	5.9 ± 0.14	5.00 ± 0.08	3.8 ± 0.12	24.00 *	35.59 *
	Autumn	7.00 ± 0.13	4.91 ± 0.09	4.33 ± 0.11	11.81 *	38.14 *
	Winter	5.50 ± 0.09	4.9 ± 0.1	4.40 ± 0.09	10.20 **	20.00 **
	Summer	10.82 ± 0.16	6.93 ± 0.13	<2.66 ± 0.04	61.61 *	75.42 *

Note: PI: Primary Influent; SE: Secondary Effluent; TE: Tertiary Effluent. TRE: Tertiary Removal Efficiency (SE to TE); ORE: Overall Removal Efficiency (PI to TE). Significance levels compared to summer peak performance: ** $p < 0.01$ (highly significant), * $p < 0.05$ (significant).

A significant seasonal variation in triclosan removal was observed. Triclosan removal peaked in the summer, in both WWTPs, with overall removal efficiencies (ORE) reaching 66.8% (plant A) and 75.4% (plant B). However, a highly significant decline ($p < 0.01$) was observed in winter, where TRE plummeted to 7.45% (plant A) and 10.2% (plant B). The drastic reduction in plant performance during winter resulted in tertiary effluent (TE) concentrations (up to 4.72 µg/L) that is over 90 times higher than the PNEC value of 0.05 µg/L. The consistent reductions observed from influent to tertiary effluents in both plants indicates triclosan removal during the wastewater treatment processes, as similarly reported by [15]. However, the 20–75.42% ORE contrasted with the 97.6–98.6% observed by [35]. The observed highly significant decrease in ORE ($p < 0.01$) during winter aligns with the seasonal trends observed by [37]**,** who reported that the elimination of pharmaceuticals in wastewater effluents was significantly inhibited during the winter in municipal systems. This inhibition may be influenced by increased sorption at lower temperatures and the temperature-induced slower microbial desorption rates. It could also result from changes in wastewater composition, and operational process conditions within the treatment systems. The superior ORE observed in plant B can be attributed to its more complex multi-stage treatment involving sand-filtration and chemical phosphorous removal via waste pickle liquor, unlike plant A which relies on oxygen-aerated activated sludge and biological nutrient removals (BNRs) [24,25]. The sand-filtration step in plant B provide an additional physical barrier that could possibly sequesters particle-bound triclosan which would otherwise have escaped secondary treatment. This study aligns with the observation by [38], who observed that plants with higher solids retention capacity and chemical-aided settling demonstrated greater reduction in triclosan content compared to those utilizing conventional biological methods. It is also in accordance with the findings of [15] who opined that changes in sludge treatment procedures and hydraulic retention durations can influence the capacity of WWTPs to eliminate triclosan.

3.3. Seasonal Dynamics and Characterization of Wastewater Physicochemical Parameters

The physicochemical data showed that the PI, SE, and TE obtained from both WWTPs exhibited significant temporal variability (Figures 5–7). These fluctuations, rather than being merely environmental indicators, actually serve as primary drivers for pollutant removal [37]. The temporal variations in physicochemical properties are similar to the observations made by [39], who also observed variability in physicochemical properties of influents and effluents obtained from some South African WWTPs.

The results also show that pH of the PI (Figure 5) was relatively stable across all seasons, fluctuating narrowly from slightly acidic to slightly alkaline (6.28 to 7.93). Since the pH range of the PI remained below the pKa of triclosan (8.14) [40]. The molecule existed primarily in its neutral, hydrophobic form. This speciation favored the high sorption rates observed in the secondary stages of both plants.

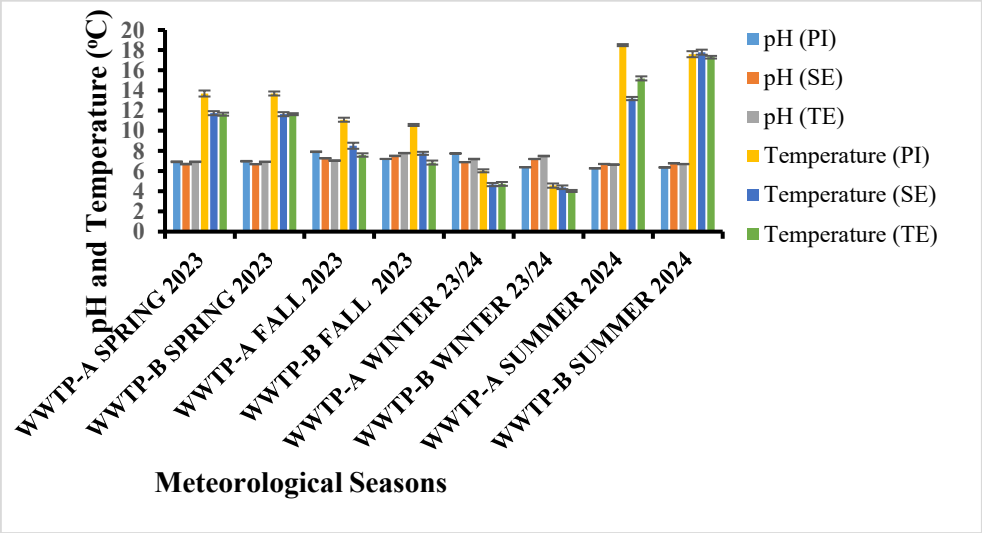

Figure 5. Variations in pH and temperature across treatment stages in two WWTPs.

The moderate negative coefficient of determination (r = −0.58 for ORE and −0.62 for *TRE*; p = 0.131) (Table 4) indicates that as the pH increases towards the alkaline range, a large fraction of the triclosan exists in its phenolate anionic form. This anionic triclosan is less susceptible to oxidation and is more hydrophilic. However, the lack of strong correlation (r > 0.80) indicates that within the observed operational range (6.28–7.93), the tertiary treatment process operated within a stable buffering range and that pH-induced changes in triclosan speciation was not the primary driver of treatment failure. The pH range (6.28–7.93) observed in this study aligns with the 6.72–7.07 observed by [39] in wastewater influents from three South African WWTPs. The stable pH levels, predominantly around pH 7, observed in the tertiary effluent indicates successful neutralization. This is also in agreement with the findings by [39].

Table 4. Pearson correlation coefficients (r) for pooled wastewater physicochemical parameters and triclosan removal efficiencies.

Physicochemical Parameter	ORE (%)	*p*-Value (ORE)	TRE (%)	*p*-Value (TRE)
Temperature (°C)	0.86	0.006 **	0.91	0.002 **
TDS (mg/L)	0.21	0.617	0.15	0.723
EC (µS/cm)	−0.38	0.353	−0.44	0.275
pH	−0.58	0.131	−0.62	0.101
Salinity (ppm)	−0.66	0.075	−0.68	0.063

** Correlation is significant at the 0.01 level.

The temperature of the PI followed a predictable seasonal trend, peaking in summer (17.6–18.5 °C) and reaching minimum in winter (4.57 °C −6.03 °C) (Figure 5). The highly significant positive correlation between temperature and TRE (r = 0.91, p < 0.01) (Table 4) possibly indicates that the kinetics of triclosan degradation are highly temperature-dependent. Higher summer temperatures likely accelerate the metabolism of microbes in the activated sludge during the secondary stage, which enhances degradation process in the tertiary treatment process. In contrast, lower winter temperatures may inhibit microbial degradation. This result aligned with that observed by [37,41].

Salinity (Figure 6) was consistently low (about 0.42 to 1.22 ppm). This probably indicates that both wastewater treatment plants mainly process freshwater-based municipal sewage, with very little or no saline intrusion. A marginally significant negative correlation (−0.68) was observed between salinity and TRE suggesting that as the ionic strength of the SE increases, particularly in winter, the efficiency of the tertiary treatment process consistently diminishes. This result contrasts with the 0.27–0.34 ppm observed by [39] in wastewater influents from three South African WWTPs.

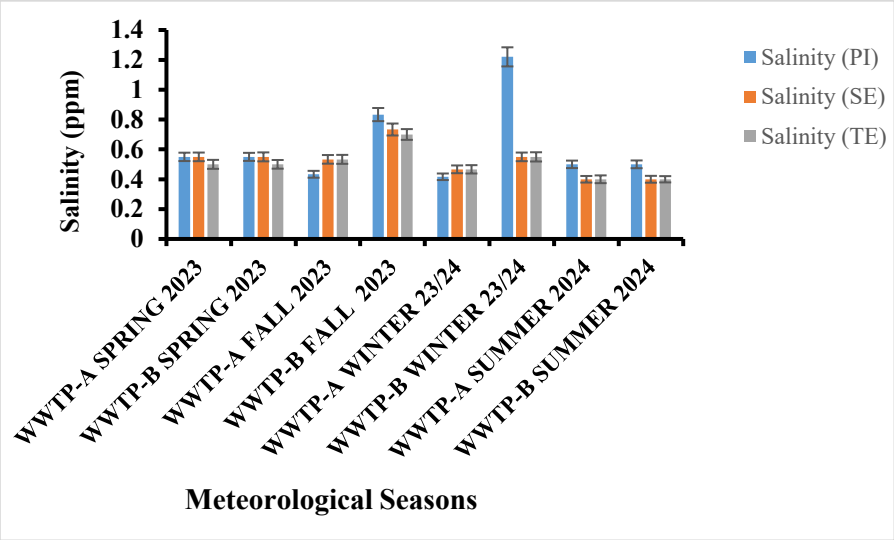

Figure 6. Variations in salinity across treatment stages in two WWTPs.

The mineral loading of the wastewater, represented by the EC and TDS, showed that the EC (894–1369 µS/cm) and TDS (603–787 mg/L) (Figure 7) typically remain elevated and consistent, increasing from PI to SE. This result aligned with that observed by [42], who observed that municipal wastewaters frequently exhibit a post-treatment rise in conductivity attributable to the transformation of organic particles into inorganic salts inside the aeration basin.

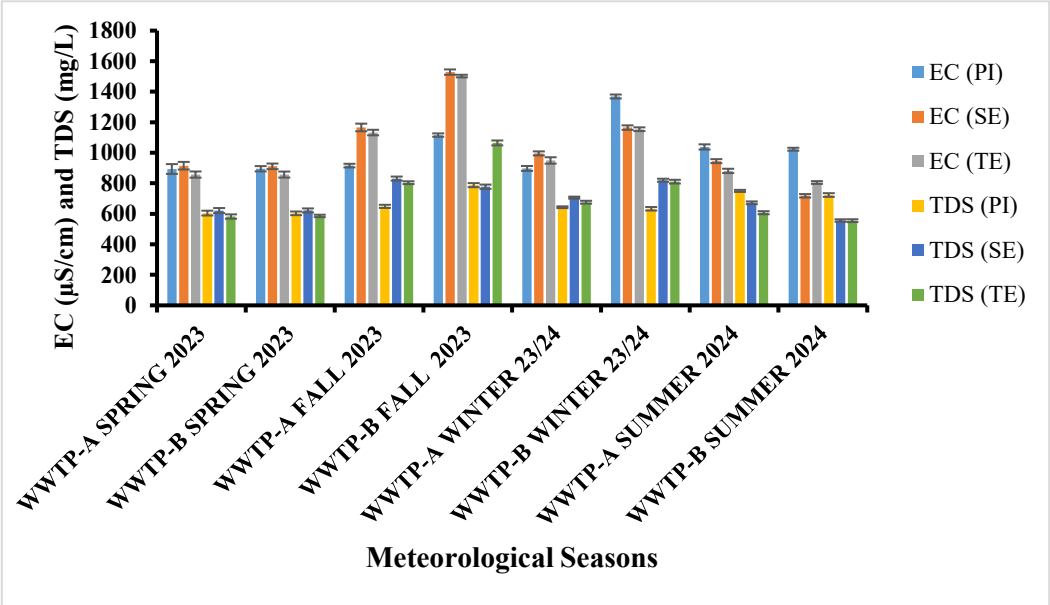

Figure 7. Variations in EC and TDS across treatment stages in two WWTPs.

The high TDS (632.8–643 mg/L) and EC values recorded in PI, particularly during winter, indicate elevated concentrations of dissolved ions such as sulfates and bicarbonates. Previous studies have suggested that increased ionic strength may influence triclosan behaviour in wastewater by altering its partitioning characteristics and reducing the apparent solubility of the neutral triclosan molecule through modification of its hydration shell [35]. He [43] further reported that reduced triclosan solubility may increase the thermodynamic driving force for partitioning from the aqueous phase to the organic fraction of activated sludge, potentially enhancing sorption even when biological degradation is thermally inhibited. Conversely, elevated ionic strength has also been reported to suppress microbial activity, alter triclosan sorption dynamics, and reduce biodegradation efficiency within activated sludge systems [35]. These contrasting mechanisms highlight the complex influence that ionic strength may exert on triclosan fate in wastewater treatment.

However, the correlation analysis performed in the present study showed that neither TDS ($p = 0.617$) nor EC ($p = 0.353$) was significantly associated with triclosan removal efficiency across the two WWTPs. Although EC exhibited a moderate negative correlation with TRE ($r = -0.44$), this relationship was not statistically

significant and therefore should not be interpreted as evidence of a causal effect. Consequently, the potential influence of elevated ionic strength on the reduced winter triclosan removal observed in this study should be regarded as a plausible mechanistic hypothesis based on previous literature rather than a statistically supported conclusion from the present dataset. Additional studies with larger sample sizes and direct measurements of ionic composition and oxidation processes are needed to verify this mechanism. The seasonal reduction in triclosan removal observed in this study is qualitatively consistent with previous reports of reduced pharmaceutical removal during winter in boreal wastewater treatment systems [37]. However, the present data do not permit attribution of this trend specifically to changes in ionic strength.

The result observed in this study is much higher than the 273–343 mg/L TDS and 546–688 µS/cm EC observed by [39]. The TDS observed in this study contrasts with the 875–2850 mg/L observed by [44], however the EC is much lower than the 190–2840 µS/cm observed by [45] in urban wastewater collected from WWTPs in France. The elevated readings in WWTP-B's influent, particularly during colder months, possibly indicate seasonal variations in residential water source attributes, groundwater infiltration, or non-industrial high-TDS domestic discharges unique to its catchment area.

3.4. Environmental Fate of Triclosan

The observed fluctuations in triclosan removal across the two wastewater treatment facilities are not merely functions of treatment stage efficiency but reflect the complex, multi-phasic environmental fate of this hydrophobic biocide. A holistic system understanding showed that the observed reduction in triclosan content of the aqueous phase is a competitive interplay between physical partitioning, microbial transformation, and chemical oxidation.

In wastewater systems with pH values below the pKa of triclosan, the compound predominantly exists in its neutral (protonated) and highly hydrophobic form due to its high K_{ow}. Within activated sludge processes, this hydrophobicity promotes preferential sorption of triclosan onto the organic fraction of sludge solids. Consequently, triclosan removal during secondary treatment may occur not only through biodegradation but also through partitioning into the sludge phase, potentially resulting in substantial solid-phase accumulation. The findings of this study showed that ORE was consistently higher than TRE thereby supporting the sludge sink theory. This observation aligns with the observations of [38,40] that up to 90% of triclosan removal in conventional wastewater treatment systems can be attributed to sorption rather than mineralization.

During winter, elevated total dissolved solids (TDS) and electrical conductivity (EC) may have influenced triclosan partitioning and sorption behavior, potentially contributing to marginal increases in solid-phase association. However, reduced microbial activity and biodegradation efficiency under lower temperatures may have limited triclosan transformation, resulting in higher residual concentrations of dissolved and particle-associated triclosan entering the tertiary treatment stage. This combination may partially explain the elevated triclosan concentrations observed in tertiary effluents during winter.

The observed reduction in triclosan concentration between the primary influent and subsequent treatment stages is likely associated with microbial transformation and sorption processes occurring during wastewater treatment. In this study, aqueous triclosan concentrations decreased from influent (5.2–10.82 µg/L) to effluent (<2.66–4.72 µg/L) phases, representing a 20–75% reduction in concentration. In the absence of direct sludge-phase measurements and operational flow-normalization data, a rigorous quantitative mass balance cannot be established. However, the mechanistic pathways driving this reduction can be inferred through triclosan's physicochemical properties and the specific configurations of the studied WWTPs. Due to its hydrophobic nature ($\log K_{ow} \approx 4.8$), a significant fraction of influent triclosan inherently partitions onto primary and secondary settled sludge via physical sorption. Concurrently, biological and chemical transformation pathways compete with this phase-partitioning. Microbially mediated processes in secondary aeration tanks can trigger hydroxylation or biomethylation, converting an estimated 2–5% of the influent burden into persistent derivatives like methyl-triclosan [46,47]. Most notably, because both monitored facilities utilize a tertiary chlorination-dechlorination sequence, chemical transformation represents a highly probable fate for the remaining dissolved fraction. The introduction of chlorine disinfectants facilitates electrophilic substitution reactions, potentially yielding chlorinated phenoxyphenols, chlorophenols, and chloroform [48,49]. Ultimately, while the observed concentration drop reflects a combined matrix of sludge partitioning and chemical degradation, future research utilizing simultaneous sludge-phase sampling and mass-flow tracking is required to definitively resolve the precise mass balance of triclosan across these treatment barriers

The triclosan removal observed in this study may therefore, in some instances, represent the conversion of triclosan into these undetectable transformation products rather than complete mineralization. In other words, the

fact that triclosan disappearance does not necessarily equate to reduction in effluent toxicity highlights a critical limitation of conventional wastewater treatment.

3.5. Environmental Risk Assessment and Regulatory Implications

The two wastewater treatment plants (WWTPs) achieved appreciable triclosan removal, with reductions of approximately 75% in WWTP-B and 67% in WWTP-A. However, the persistence of quantifiable triclosan concentrations in the treated effluents indicates that conventional wastewater treatment processes did not achieve complete removal of this hydrophobic contaminant. Olaniyan et al. [50] reported that inadequate triclosan removal from wastewater may result in its accumulation in aquatic organisms and sediments, thereby posing potential risks to aquatic ecosystems and human health. Based on the quantifiable tertiary effluent concentrations (2.66–4.72 µg/L), triclosan levels substantially exceeded the adopted Predicted No-Effect Concentration (PNEC) of 0.05 µg/L for aquatic environments [51], with corresponding risk quotient (RQ) values reaching 94.4, indicating considerable ecological concern for the measured samples. However, because the analytical method had a limit of quantification (LOQ) of 2.66 µg/L, ecological risks associated with concentrations below the LOQ could not be excluded. The measured triclosan concentrations also exceeded the Minnesota Department of Health's drinking water guidance value of 0.05 µg/L [52], highlighting the need for continued monitoring and improved treatment strategies.

The measured environmental concentration (MEC) and the predicted no-effect concentration (PNEC) were used to assess the potential ecological risk of triclosan. The resulting risk quotient (RQ) values (Table 5) ranged from 53.2 (summer, WWTP-B) to 94.4 (winter, WWTP-A), substantially exceeding the threshold for high ecological risk across all sampling seasons. These findings indicate that quantifiable triclosan concentrations in tertiary effluents may pose a considerable risk to sensitive aquatic primary producers, particularly green algae, which form the foundation of aquatic food webs. However, because the analytical method could not reliably quantify triclosan below the LOQ, ecological risks associated with lower concentrations could not be excluded.

The ecological risk assessment based on quantifiable triclosan concentrations revealed high risk quotient (RQ) values ranging from 53.2 to 94.4 (Table 5), indicating considerable ecological risk for the monitored tertiary effluent samples. These values are comparable with the RQ range of 20–327 for algae reported by Mohan and Balakrishnan [53] for wastewater effluents in India under low-dilution conditions, although they are lower than the maximum RQ of 600 reported by Gumbi et al. [54] for South African wastewater effluents. The elevated RQ values observed in the present study are consistent with the broader regulatory concern surrounding triclosan, which contributed to its prioritization under the European Union Water Framework Directive because of its persistence, bioaccumulation potential, and endocrine-disrupting properties [55], followed by subsequent restrictions on its use in cosmetic products in October 2025 [56].

The elevated RQ values may be attributed to the relatively high measured environmental concentrations (MECs), the conservative PNEC adopted for algae, and the incomplete removal of triclosan by conventional biological wastewater treatment processes. The lack of advanced polishing technologies, such as ozonation or granular activated carbon, may also have contributed to the persistence of triclosan in tertiary effluents. These findings suggest that, although the investigated wastewater treatment plants achieved substantial triclosan removal, quantifiable residual triclosan concentrations in treated effluents may still pose considerable ecological risk to sensitive aquatic ecosystems receiving wastewater discharges.

However, because the analytical method had a limit of quantification (LOQ) of 2.66 µg/L, it could not reliably quantify triclosan at concentrations approaching the adopted PNEC (0.05 µg/L). Consequently, while the measured concentrations clearly indicate high ecological risk for the quantified samples, potential ecological risks associated with triclosan concentrations below the LOQ cannot be excluded. Future monitoring using more sensitive analytical techniques and the implementation of advanced polishing processes are therefore recommended to improve environmental risk characterization and further reduce triclosan discharge.

Table 5. Seasonal Risk Quotients (RQ) for Tertiary Effluents.

Plant	Season	MEC (µg/L)	PNEC (µg/L)	RQ	Risk Level
WWTP-A	Spring	3.50	0.05	70	High
	Autumn	4.60	0.05	92	High
	Winter	4.72	0.05	94.4	High
	Summer	3.10	0.05	62	High
WWTP-B	Spring	3.80	0.05	76	High
	Autumn	4.33	0.05	86.6	High
	Winter	4.40	0.05	88	High
	Summer	<2.66	0.05	53.2	High

4. Limitations and Future Recommendations

Although grab sampling was employed to preserve triclosan stability and minimize sorption-related losses associated with prolonged composite sampling, the approach may not fully capture short-term temporal fluctuations in triclosan concentrations and treatment performance within the wastewater treatment systems.

In addition, removal efficiencies were based on simultaneous influent–effluent sampling and therefore may not fully reflect hydraulic retention dynamics. The absence of continuous flow and operational process data also limited comprehensive mass balance evaluation.

Furthermore, although the SPE-coupled spectrophotometric method demonstrated good linearity, recovery, and reproducibility within the concentration range investigated, its LOQ remained higher than the adopted PNEC value, limiting the detection of environmentally relevant ultra-trace triclosan concentrations that are routinely measurable using LC-MS/MS.

Future studies should incorporate high-frequency flow-proportional sampling, hydraulic retention time assessment, continuous operational process monitoring, and advanced analytical techniques (e.g., LC-MS/MS) with lower detection limits to improve temporal characterization, contaminant mass balance evaluation, and ecological risk assessment in wastewater treatment systems.

5. Conclusions

The physicochemical characteristics of both wastewater treatment plants were generally consistent with municipal wastewater treatment systems; however, seasonal fluctuations were associated with variations in triclosan removal performance. Substantial triclosan reduction was observed across the treatment trains, although elevated residual concentrations persisted in tertiary effluents during certain seasons, particularly winter. Correlation analysis identified temperature as the principal factor associated with seasonal variations in triclosan removal, whereas the proposed influence of ionic-strength-related parameters (TDS and EC) should be regarded as a plausible mechanistic hypothesis rather than a statistically supported conclusion.

Ecological risk assessment based on quantifiable triclosan concentrations in tertiary effluents indicated that the measured concentrations exceeded the adopted PNEC of 0.05 μg/L, RQ values reaching 94.4, indicating considerable ecological concern for the monitored samples. However, because the analytical method had LOQ of 2.66 μg/L, it could not reliably quantify triclosan at concentrations approaching the PNEC. Consequently, while the present findings clearly demonstrate ecological risks associated with samples containing triclosan above the LOQ, they do not exclude the possibility of ecological risks associated with concentrations below the quantification limit. Future studies employing more sensitive analytical techniques, such as LC-MS/MS, are therefore recommended to improve the detection of trace triclosan concentrations and enable more refined ecological risk assessments. Consequently, optimization of tertiary treatment processes and consideration of advanced polishing technologies, such as granular activated carbon or ozonation, may further reduce triclosan release and enhance protection of receiving aquatic ecosystems.

6. Recommendation

In order to minimize triclosan content of wastewater, there is a need to further purify the wastewater effluents using other techniques such as GAC, ozonation, carbon adsorption, or other emerging remediation techniques such as nanotechnology.

Author Contributions

M.O.Y.: conceptualization, methodology, software; M.O.Y.: data curation, writing—original draft preparation; M.O.Y. and O.J.O.: visualization, investigation; S.K.P.: supervision; S.K.P. and S.S.: software, validation; O.J.O. and S.S: writing—reviewing and editing. All authors have read and agreed to the published version of the manuscript.

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Not applicable.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Conflicts of Interest

The authors declare no conflicts of interest.

Use of AI and AI-Assisted Technologies

During the preparation of this work, the authors used Quillbot to enhance the clarity, tone, and style of the scientific writing. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the content of the published article."

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