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Removal of pharmaceuticals with ozone at 10 Swedish wastewater treatment plants

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Abstract

Pilot-scale tests were run with ozonation for reduction of 24 pharmaceuticals at 10 full-scale wastewater treatment plants in southern Sweden. Reduction was evaluated based on doses of 3, 5 and 7 g O_3/m^3 at all plants. The reduction of pharmaceuticals reached on average 65% at 3 g O_3/m^3, 78% at 5 g O_3/m^3 and 88% for 7 g O_3/m^3 in terms of total concentration of pharmaceuticals. Specific ozone dose (ratio O_3:TOC) was found to be highly influential on pharmaceutical removal. At two WWTPs, the pharmaceutical removal was severely reduced.

Key words: ozonation, pharmaceuticals, pilot-scale

INTRODUCTION

Many countries are considering the need for reduction of pharmaceuticals and other organic micropollutants in wastewater. In Switzerland the legal framework is already in place (Eggen *et al.* 2014), in the EU, the list of priority pollutants already include organic micropollutants and the 'watch list' has recently been extended with a number of pharmaceuticals (2013/39/EU). In Sweden, the first full-scale installation based on ozonation, is under construction (IVL 2016) even though the needs and requirements of such a treatment step are still debated.

Ozonation and activated carbon treatment, or a combination seems to be the winning technologies for reduction of organic micropollutants. Full-scale installations have only been reported in a few countries (Cimbritz *et al.* 2016) but pilot-scale installations have been running at several places in order to test the technology and to give guidelines for design (Hollender *et al.* 2009; Wert *et al.* 2009; Ibáñez *et al.* 2013; Margot *et al.* 2013). Such guidelines are problematic as long as the substances included in the control program and the limits and control methods are not selected at the same time. Typically, a number of substances in high concentration for which reasonable analytical methods exist are selected and the final effluent concentration or the percentage reduction is used as evaluation criteria (Huber *et al.* 2005; Hansen *et al.* 2010; Antoniou *et al.* 2013).

For design of equipment and estimation of the economy in ozonation, guidelines for the needed ozone dose is typically based on the content of organic material in the treated wastewater. As the major part of the ozone is consumed by organic matter left after the normal treatment only a minor part is used to oxidize micropollutants. In addition, pH, alkalinity and a number of substances that might be present in treated wastewater are known to have a significant impact on ozone

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consumption and consequently on the ozone dose needed (Gottschalk *et al.* 2010; Hansen *et al.* 2010; Antoniou *et al.* 2013; Hey *et al.* 2014).

The Swedish debate about the need for reduction of organic micropollutants, especially pharmaceuticals, might end up with a general requirement of wastewater treatment plants to include reduction of pharmaceuticals in the near future. Consequently, a deeper understanding of the expected need for ozone addition is required before the economic and environmental consequences can be evaluated. Testing at single treatment plants might not be representative for other WWTPs. Therefore, the main objective of the present study was to test whether ozone can be used in the same manner and reach comparable results in terms of pharmaceutical reduction regardless of the WWTP configuration. The secondary objective was to study how the concentration of TOC in the treated wastewater impacts the efficiency of pharmaceutical reduction and evaluate whether it can be used in a general model to control the amount of ozone being produced in a full-scale ozone installation.

MATERIALS AND METHODS

Pilot plant

A schematic representation of the ozone equipment used throughout the trials is depicted in Figure 1. The objective of the system was to produce and dissolve ozone into wastewater in a measurable and repeatable way at 10 WWTPs. All equipment was housed in a 20 feet container. A submerged centrifugal pump delivered 18–20 m^3/h of treated wastewater into a drum filter. The purpose of the drum filter was to reduce turbidity and minimize the impact of fluctuations in WWTP performance (such as an underperforming clarifier) on the ozone pilot plant. The flow entering the drum filter was monitored by a flow meter at the inlet. The reduction of turbidity across the filter was monitored by two turbidity meters positioned before and after the filter. Filtered wastewater entered a holding tank to equalize the flow before the ozone injection. The excess flow (about 12 m^3/h) was discharged back into the wastewater stream, downstream of the submerged centrifugal pump.

Ozone was produced from onsite generated oxygen. A main PLC was connected to the flow meter, ozone generator and ozone concentration meter, which enabled the ozone dose (displayed as g O_3/m^3 on the main PLC screen) to be monitored manually throughout the trials. The wastewater

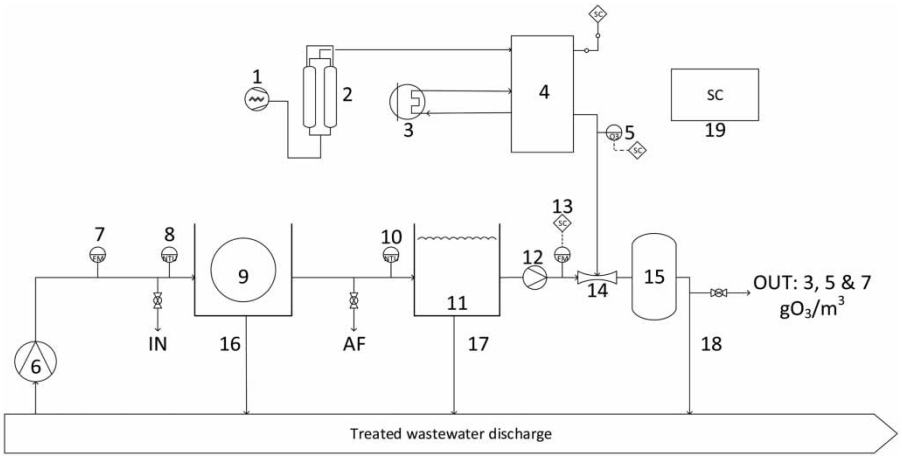

Figure 1 | Schematic overview of the equipment used. 1: Compressor (AirSep, Topaz Plus), 2: PSA oxygen supply (AirSep, Topaz Plus), 3: Chiller (Lauda, UC Mini), 4: Ozone generator (Primozone, GM2), 5: Ozone concentration meter (BMT, 964-C), 6: Submerged centrifugal pump (Mecana, TF2), 7: Flowmeter (Mecana, TF2), 8: Turbidity meter (Mecana, TF2), 9: Drum filter (Mecana, TF2), 10: Turbidity meter (Mecana, TF2), 11: 1 m^3 equalization tank, 12: Booster pump (Grundfos, CM5-4), 13: Flow meter (Honsberg), 14: Venturi injector (Mazzei, 1583), 15: 500 l pressurized reaction vessel (HRT: 5 min), 16: Sludge from drum filter, 17: Excess flow, 18: Discharge from ozonation, 19: Main PLC (Schneider, Modicon M251).

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from the equalization tank (6 m^3/h) was pumped with a booster pump through a venturi injector and mixed with the ozone, the flow of water being monitored by a flow meter. The ozonated wastewater then entered a pressurized reaction tank (5 min HRT at 6 m^3/h) before being discharged back into the wastewater stream downstream of the submerged centrifugal pump. There were a total of 3 sampling locations, at the inlet of the drum filter (IN), after the filter (AF) and outlet (OUT) of the pressurized reaction tank.

Operation of the pilot plant

The trials were run in the same manner throughout all 10 WWTPs. The submerged pump was lowered into the discharge stream at the WWTP, wastewater was pumped through the system for at least 24 hours prior to the commencement of the trials. This was done to safeguard that no residuals from the previous trial were present.

The ozone production was started and adjusted manually until the production corresponded to the lowest ozone dose (3 g O_3/m^3 wastewater). After the ozone flow had reached the required dosage, it was kept running for 20 minutes (4× HRT) before the first samples were taken. Samples were collected in glass bottles from points IN (0.5 L), AF (0.5 L) and OUT (1.5 L) every 10 minutes for a total of 60 minutes which resulted in 3.5 L sample from point IN, 3.5 L from point AF and 10.5 L from point OUT (3 g O_3/m^3). The next ozone doses (5 and 7 g O_3/m^3 wastewater) were then introduced to the system in the same manner. The samples from points IN and AF were taken as composite samples for the entire trial run.

Analysis

All samples were analyzed in the lab at Lund University. The chemical analysis was conducted with a spectrophotometer (Hach-Lange DR 2800): COD (Hach-Lange, LCK 314), TOC (LCK 385), Tot-P (LCK 349), PO_4-P (LCK 349), Tot-N (LCK 138), NH_4-N (LCK 303), NO_3-N (LCK 339), NO_2-N (LCK 341). The other analysis were conducted using the standard procedures: SS (according to SS-EN 872:2005) and pH (WTW pH 320).

SUVA 254 measurement was conducted with a modification of the standard method published by USEPA (2005), absorbance was measured at 254 nm and the results were normalized with regards to TOC. The SS content in the samples taken from point OUT were so low that TOC can be regarded as dissolved organic carbon. SUVA 254 was not conducted for the first three WWTPs.

Samples were also sent to IVL (Swedish Environmental Research Institute) for pharmaceutical analysis (liquid chromatography-tandem mass spectrometry) were carried out in accordance with Gros *et al.* (2006). The pharmaceuticals analyzed are listed in Table 1.

Wastewater treatment plants

The trials were run at 10 different WWTPs in southern Sweden (Table 2), all designed for more than 10,000 PE. There are differences in the geographical location of the plants, as well as the configuration and industries connected to them. The plants are briefly described in Table 2.

Sjölunda WWTP has an unusual implementation of BOD and nitrogen removal. A detailed description of the plant can be found in (Hanner *et al.* 2003). In short, BOD is removed in a high loaded activated sludge plant. Nitrification takes place in trickling filters with plastic carriers followed by denitrification in a two-stage MBBR system. Final separation takes place in a flotation plant.

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Table 1 | The 24 pharmaceuticals included in the analysis

Name	Type	Name	Type
Amlodipine	Antihypertensive	Metoprolol	Antihypertensive
Atenolol	Antihypertensive	Naproxen	Anti-inflammatory
Bisoprolol	Antihypertensive	Oxazepam	Sedative
Caffeine	Stimulant	Paracetamol	Anti-inflammatory
Carbamazepine	Sedative	Propranolol	Antihypertensive
Ciprofloxacin	Antibiotic	Ranitidine	Antiulcer
Citalopram	Antidepressant	Sertralin	Antidepressant
Diclofenac	Anti-inflammatory	Sulfamethoxazole	Antibiotic
Furosemide	Diuretic	Terbutaline	Asthma medication
Hydrochlorothiazide	Antihypertensive	Tetracycline	Antibiotic
Ibuprofen	Anti-inflammatory	Trimetoprim	Antibiotic
Ketoprofen	Anti-inflammatory	Warfarin	Anticoagulant

Table 2 | General description of the WWTPs in this trial.

WWTP	PE (connected)	BOD removal	Nitrification	Denitrification	Sand filtration
Sternö	21,200	AS	AS	AS	X
Sjöhög	33,900	AS	AS	AS	
Nyvångsverket	11,800	TF	TF	AS	X
Torekov	12,900	AS	AS	AS	
Sjölunda	317,000	AS	TF	MBBR	
Källby	98,600	AS	AS	AS	
Ellinge	20,100	AS	AS	AS	
Kävlinge	29,000	AS	AS	AS	X
Svedala	12,000	AS	AS	AS	
Västra Stranden	70,000	AS	AS	AS	

AS: activated sludge, TF: trickling filter, MBBR: moving bed biofilm reactor. X in the final column denotes that the WWTP utilizes a sand filter

RESULTS AND DISCUSSION

Pharmaceuticals

There are at present no set concentration limits for specific pharmaceuticals within the EU. Therefore, the reduction of pharmaceuticals in this paper is presented as reduction of the total concentration of all analyzed pharmaceuticals. Furthermore, as there are no reduction criteria in use in the EU, the Swiss limit (Eggen *et al.* 2014) of 80% reduction of pharmaceuticals is designated as the target value.

The sum of all 24 pharmaceuticals is depicted in Table 3. If a pharmaceutical concentration was below the detection limit, the concentration of such compound was set to half the detection limit. The total concentration of pharmaceuticals that entered the pilot plant varied between 4,600 and 18,700 ng/L. As the analysis for pharmaceuticals were conducted on dissolved compounds only and no precipitating agents were employed, the drum filter is not considered to have had any impact on the pharmaceutical removal in these trials.

Figure 2 shows the total pharmaceutical removal at the three applied ozone doses. On average, the total concentration of measured pharmaceuticals was reduced by 65% at 3 g O_3/m^3, 78% at 5 g O_3/m^3

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Table 3 | Sum of the pharmaceutical concentrations (ng/L) at points IN and OUT at the 10 WWTPs.

		OUT		
		Ozone dose		
WWTP	IN	3 g O_3/m^3	5 g O_3/m^3	7 g O_3/m^3
Sternö	10,651	5,064	2,505	1,093
Sjöhög	15,321	9,270	3,487	1,418
Nyvångsverket	8,359	2,284	1,597	674
Torekov	4,603	1,223	458	240
Sjölunda	12,420	2,969	2,027	860
Källby	12,095	2,308	1,048	711
Ellinge	17,860	6,680	5,040	1,953
Kävlinge	10,896	2,253	640	472
Svedala	18,702	10,189	10,258	6,283
Västra Stranden	7,838	2,453	2,156	1,683

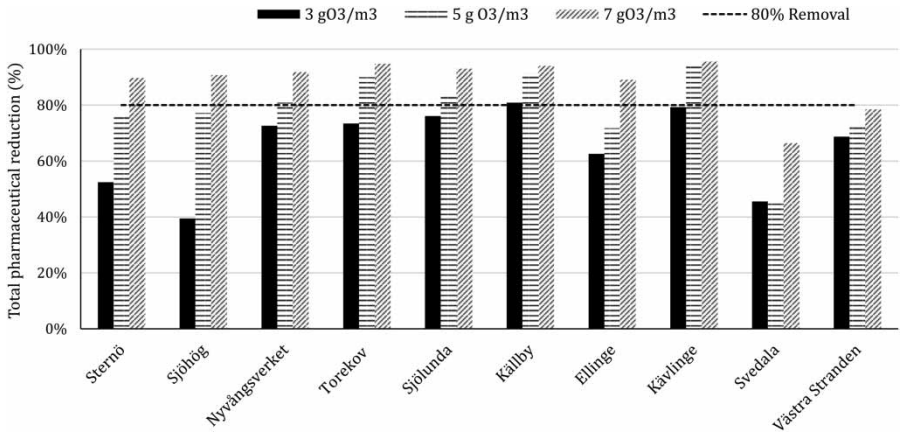

Figure 2 | Total pharmaceutical removal at the three ozone doses (3, 5 and 7 g O_3/m^3).

and 88% at 7 g O_3/m^3. However, these figures are the average removal from all the WWTPs, if the last two WWTPs were to be removed from consideration, said figures reaches 67%, 83% and 92% for 3, 5 and 7 g O_3/m^3 doses. This increase in averages points to the impact of the lower removal at Svedala and Västra Stranden and elicits further analysis, as something clearly caused the pharmaceutical removal to be less effective at those WWTPs. In the case of Svedala WWTP, the total concentration of pharmaceuticals entering the pilot plant reached 18,702 ng/L which is the highest of all the WWTPs, however, it is considered to be comparable to Ellinge WWTP at 17,860 ng/L. Furthermore, the inlet concentration to the pilot plant at Västra Stranden (7,838 ng/L) is comparable to Nyvångsverket WWTP (8,359 ng/L).

Thus, the inlet concentrations of pharmaceuticals at Svedala and Västra Stranden are not considered to be responsible for the lower reduction at those plants. When applying the criteria set for removal of pharmaceuticals (>80%), 3 g O_3/m^3 is sufficient only at two WWTPs (Källby and Kävlinge). At 5 g O_3/m^3 the number of plants reaching the criteria increases to 5 (Nyvång, Torekov, Sjölunda, Källby and Kävlinge). At 7 g O_3/m^3 all but the last two WWTPs reaches 80% removal.

The pharmaceutical compounds are divided into groups in Table 4. The compounds which were removed above 80% at more than 6 WWTPs are grouped according to the ozone dose required to reach that criteria. The column 'Removed <80% or at <6 plants' contains the pharmaceuticals

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Table 4 | Individual pharmaceuticals divided into groups depending on to which extent they were removed.

Removed at 3 g O_3/m^3	Removed at 5 g O_3/m^3	Removed at 7 g O_3/m^3	Removed <80% or at <6 plants	Not found in sufficient concentration
Diclofenac	Citalopram	Hydrochlorothiazide	Ibuprofen	Ciprofloxacin
Furosemide		Sulfamethoxazole	Warfarin	Tetracycline
Naproxen		Atenolol	Caffeine	Amlodipine
Carbamazepine		Bisoprolol	Ketoprofen	
Propranolol		Metoprolol	Oxazepam	
Ranitidine		Sertralin	Paracetamol	
			Terbutaline	
			Trimetoprim	
33%	**2.8%**	**36.7%**	**26.5%**	**1.5%**

which were not removed above 80% or removed above 80% but in fewer than 6 WWTPs and are considered difficult to remove. The last column contain the pharmaceuticals that were not found in sufficient concentration at sufficient number of WWTPs. The average relative inlet concentration of the compound groups (%) are listed at the bottom of each group.

The first two groups in Table 4 ('Removed at 3 g O_3/m^3' and 'Removed at 5 g O_3/m^3') corresponds reasonably well with the findings of Antoniou *et al.* (2013) and Margot *et al.* (2013). The last two groups ('Removed at 7 g O_3/m^3' and 'Removed <80% or at <6 plants') corresponds well with the findings of Hey *et al.* (2014) and Hollender *et al.* (2009).

The average removal of individual pharmaceutical compounds is depicted in Figure 3 along with the standard deviation of each compound. As ciproflaxin, tetracycline and amlodipine were only found in a handful of WWTPs they are excluded from the graph.

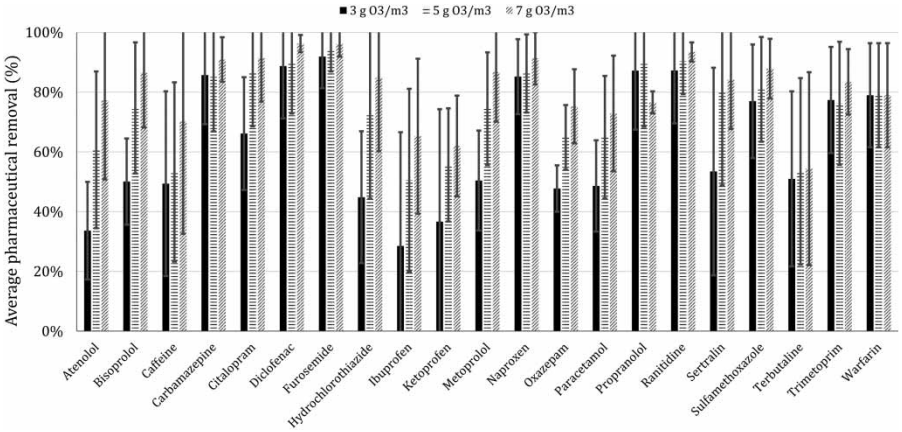

Figure 3 | Average pharmaceutical removal of individual pharmaceutical compounds at all 10 WWTPs.

As is apparent from Figure 3, the standard deviation is quite large for some of the pharmaceutical compounds, especially at the lowest ozone dose (3 g O_3/m^3). For instance, ibuprofen exhibits a standard deviation of 38% at the lowest ozone dose. The reason for this rather large standard deviation is unknown, however, it is not surprising since the trials were conducted in pilot-scale at several WWTPs with varying wastewaters.

A radar chart of the removal of diclofenac, from the group 'Removed at 3 g O_3/m^3' in Table 4 is depicted in Figure 4. Diclofenac is removed completely at all WWTPs at the lowest ozone dose

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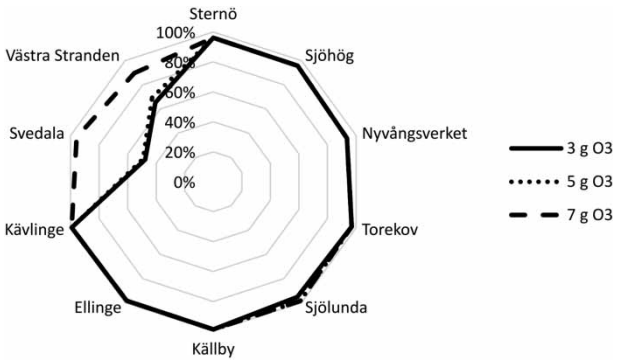

Figure 4 | Radar chart of the removal of diclofenac at 10 WWTPs.

except at Svedala and Västra Stranden, where it takes an ozone dose of 7 g O_3/m^3 to remove this compound with more than 80%.

The removal of citalopram (from the group 'Removed at 5 g O_3/m^3' in Table 4) is depicted in Figure 5. This compound was not found in Västra Stranden so that WWTP is excluded from this chart. An ozone dose of 5 g O_3/m^3 is required to remove this compound above 80% at all plants except Svedala where it is not removed above 40% at any ozone dose.

When the removal of hydrochlorothiazide (from the group 'Removed at 7 g O_3/m^3' in Table 4) is plotted in the same way (Figure 6) it becomes apparent that the more difficult a compound is to remove the larger the variations in removal becomes. For instance, in both Nyvångsverket and Torekov this compound is removed by 80% with the lowest ozone dose (3 g O_3/m^3). Whereas in Sternö, Sjöhög, Sjölunda and Ellinge the lowest ozone dose does not remove this compound to more than 40%. None of the ozone doses were sufficient to remove hydrochlorothiazide at neither Svedala nor Västra Stranden.

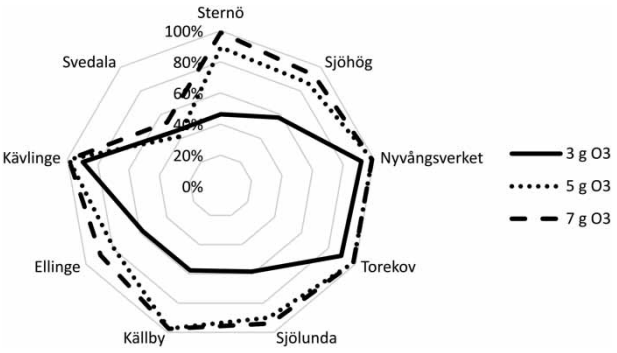

Figure 5 | Radar chart of the removal of citalopram at 9 WWTPs.

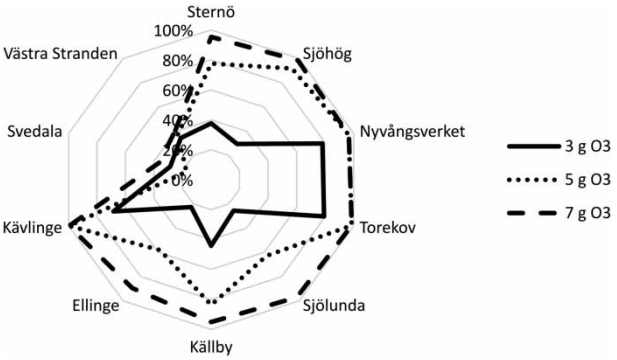

Figure 6 | Radar chart of the removal of hydrochlorothiazide of at 10 WWTPs.

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In most of the samples, the increasing dose of ozone (3 to 7 g O_3/m^3) oxidized nitrite to below the detection limit. As ozone reacts well with nitrite this was not unexpected, however, the samples from Svedala and Västra Stranden did not show the same trend and nitrite was still measurable after ozonation. The lack of nitrite oxidation at those plants indicates that ozone scavenging took place. The ingoing nitrite concentrations were so low at those plants (0.14 for Svedala and 0.09 mg/L for Västra Stranden) that the nitrite is not considered to be the reason for the poor removal of pharmaceuticals.

COD and TOC

COD was measured in all samples and ranged between 21 mg/L and 35 mg/L, but since COD analysis is not in general available in Sweden anymore TOC is used instead. The concentration of total organic carbon discharged from the WWTPs (Table 5), ranged between 8.4 and 13.9 mg TOC/L. Ozone did remove some of the TOC albeit not to a high degree.

A closer look at the TOC figures reveals that the TOC values for Svedala and Västra Stranden WWTPs are not so high as to explain the discrepancy in pharmaceutical removal at those plants.

Table 5 | TOC measurements (mg TOC/L) in points IN, AF and OUT at the 10 WWTPs.

			OUT		
			Ozone dose		
WWTP	IN (mg/L)	AF (mg/L)	3 g O_3/m^3	5 g O_3/m^3	7 g O_3/m^3
Sternö	13.9	[a]	13.9	13.5	13.4
Sjöhög	10.6	[a]	10.1	9.8	9.9
Nyvångsverket	11.4	[a]	8.1	8.1	8.1
Torekov	9.5	8.7	8.5	8.6	8.4
Sjölunda	11.0	10.8	9.3	10.6	9.9
Källby	9.8	8.9	8.1	8.0	8.2
Ellinge	13.1	13.1	12.9	12.5	12.5
Kävlinge	8.4	8.2	7.3	7.7	7.4
Svedala	13.3	12.3	12.7	12.5	12.5
Västra Stranden	11.4	11.4	10.9	10.8	11.0

[a]No results available due to lacking samples.

SUVA 254 nm

The SUVA 254 values are depicted in Figure 6. A decline in SUVA 254 can be considered to be an indicator of increasing pharmaceutical removal as well as a decline in the total concentration of aromatic substances (Wittmer *et al.* 2015). The majority of the SUVA 254 values in Figure 7 declines as the ozone dose increases. The SUVA 254 results obtained from Svedala follow the same trend as the other WWTPs, however, with a much lower reduction of aromatics at the higher ozone doses. In Västra Stranden, the SUVA 254 does not decline at all even at 7 g O_3/m^3. The behavior displayed at Västra Stranden WWTP indicates that ozone scavenging took place to a high degree.

Pharmaceutical removal as a function of TOC

The relationship between TOC and pharmaceutical removal is depicted in Figure 8. TOC and ozone dose are combined into specific ozone dose (ratio O_3:TOC).

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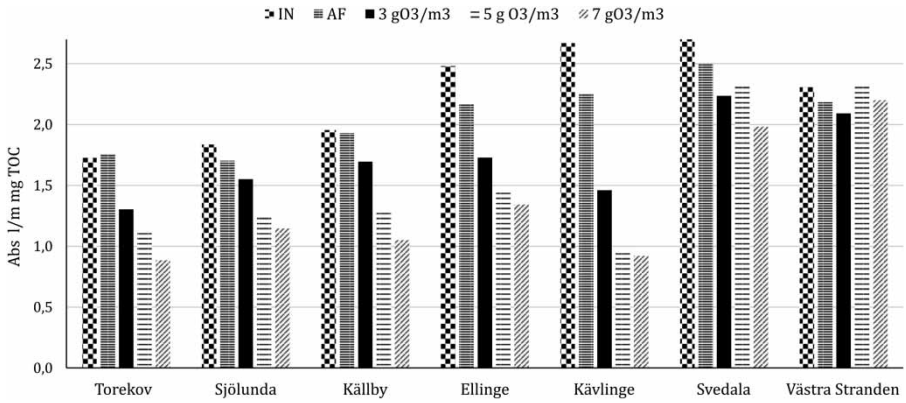

Figure 7 | The specific UV absorbance at 254 nm at the different measuring points.

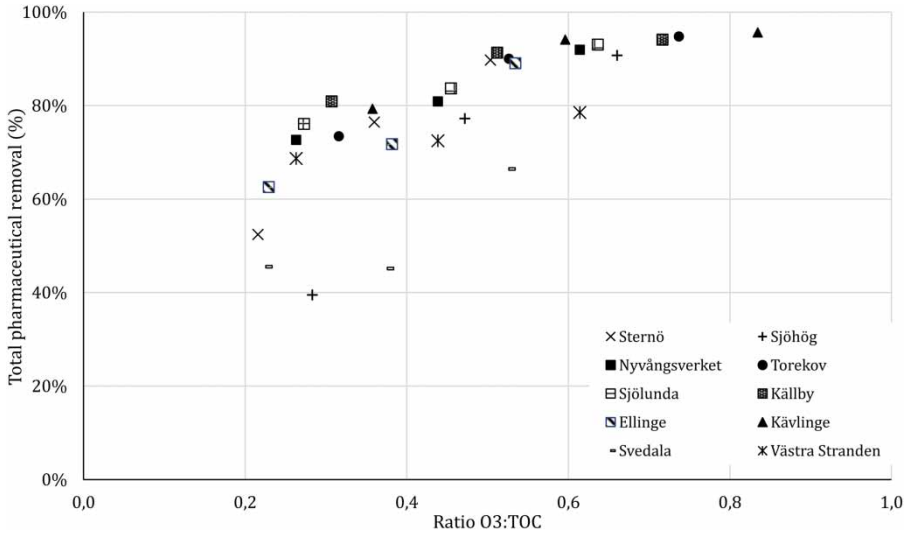

Figure 8 | A combined graph of specific ozone dose (ratio O_3:TOC) and pharmaceutical removal.

As the ratio of ozone to TOC increases so does the pharmaceutical removal. The removal efficiency increases rapidly when O_3:TOC is increased from approximately 0.2 to 0.4 after which the total removal levels off. This overall behavior is not surprising as the compounds which are easily removed (removed at 3 and 5 g O_3/m^3, Table 4) are removed well above 80% at the lower doses (O_3:TOC 0.2–0.4). Followed by the more difficult compounds (removed at 7 g O_3/m^3 and Removed <80% and/or at <6 plants, Table 4) which requires a higher specific ozone dose, eventually leaving only the compounds which are not susceptible to ozone oxidation in this range of ozone doses. The spread in the data points at the lower region (O_3:TOC 0.2–0.4) is quite substantial while being more clustered together in the higher region (O_3:TOC 0.4–0.8).

The apparent trend seen in Figure 8 can be useful in the early stages when an ozone system is to be sized. However, the high degree of spread between the data points (especially between O_3:TOC 0.2–0.4) and the low number of samples (3 samples at 10 WWTPs) points to that further testing is needed before the ratio O_3:TOC can be used as an online control parameter in full-scale. However, if tests were to be performed at one WWTP instead of 10 and run for a longer time, it is very likely that a model of how specific ozone dose impacts pharmaceutical removal could be found for that specific WWTP.

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In these trials, the only ozone dose able to meet the >80% removal criteria at a majority (8 out of 10) of the plants is 7 g O_3/m^3. A detailed cost calculation has not been made for this paper. However, a recent publication by Mulder *et al.* (2015) calculated the cost of running an ozone installation with a dose of 7.7 g O_3/m^3 to 0.16 €/m^3 treated wastewater (±0.03 €) for a 300,000 p.e. WWTP. The cost of running a comparable PAC (powdered activated carbon) installation reached 0.18 €/m^3 (±0.03 €) treated wastewater (Mulder *et al.* 2015).

Svedala and Västra Stranden WWTPs

The removal of pharmaceuticals was in general quite high when excluding the last two WWTPs. However, when the last two WWTPs are included, the averages drops significantly. The fact that nitrite was detected in the samples from these plants after ozonation was surprising since nitrite is highly reactive with ozone. Therefore, the most probable reasons for the lower pharmaceutical reduction at these plants are either an equipment failure leading to lower ozone doses or ozone scavenging of an unknown compound. No failures of the ozone equipment was detected at either of these trial runs which enhances the probability of an unknown ozone scavenging compound.

CONCLUSIONS

The main purpose of the pilot-scale trials was to evaluate the practical application of ozone at different WWTPs without considering the differences at the plants. A criteria of 80% total removal of pharmaceuticals was established as a benchmark. This criteria was met at all WWTPs at 7 g O_3/m^3 except at Svedala and Västra Stranden, therefore the process can be said to remove pharmaceuticals efficiently and with reasonably comparable results but only at the higher ozone doses. The reason for the lower removal efficiency at Svedala and Västra Stranden WWTPs was not found.

A link between specific ozone dose (ratio O_3:TOC) and pharmaceutical removal efficiency was found to exist but it is not accurate enough to be integrated as a parameter to control the output of ozone as of yet. Further work is clearly needed to acquire a general model which can be implemented at any WWTP.

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