UNIVERSITY OF CALIFORNIA

Los Angeles

Enhanced Removal of Pharmaceuticals and Personal Care Products with Increasing Sludge Retention Time in the Activated Sludge System

A thesis submitted in partial satisfaction

of the requirements for the degree Master of Science

in Civil Engineering

by

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2008

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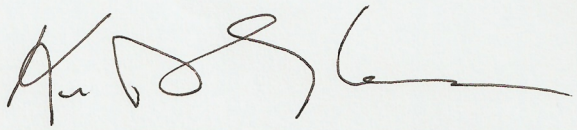

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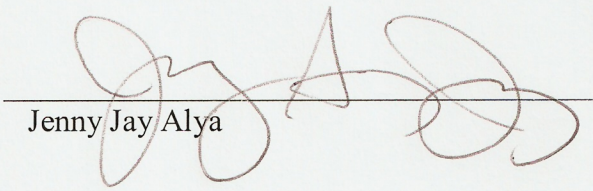

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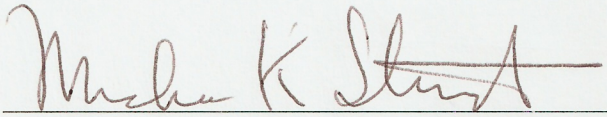

Michael K. Stenstrom, Committee Chair

University of California, Los Angeles

2008

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ABSTRACT OF THE THESIS

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Professor Michael K. Stenstrom, Chair

The issue of pharmaceuticals and personal care products (PPCPs) in the environment has been emerging for years. Many of the compounds are not fully eliminated in the wastewater treatment; they are persistent and ubiquitous in the environment. This thesis studied the nature and environmental presence of pharmaceuticals and personal care products and collected the data from the literature. The findings showed that the removal rates of most compounds investigated observed a statistical relationship with the sludge retention time (SRT), whereas carbamazepine, one of the most persistent compounds, was not eliminated during the treatment even at high SRTs. The results also showed that no significant differences between the activated sludge system and membrane bioreactor at comparable SRTs; this led to the conclusion that high removal efficiency can be

achieved in the activated sludge process and membrane bioreactor due to high SRTs rather than individual treatment technologies.

INTRODUCTION

The concern for pharmaceuticals and personal care products (PPCPs) are considered emerging contaminants in surface water due to their extensive and increasing use in human activities. Pharmaceuticals and personal care products can enter environments via different pathways such as municipal wastewater or land runoff from agricultural application. As our household wastewater makes it way through the wastewater treatment plant, some amounts of pharmaceuticals and personal care products are not fully eliminated when the treated wastewater exits the plant as effluent. While much attention and regulation regarding water pollution has focused on hazardous, highly toxic chemicals and their health effects on humans and aquatic wildlife, the increasingly widespread use of PPCPs and their effect on aquatic wildlife have been under appreciated. Many of the chemicals used in PPCPs, when present at environmentally relevant levels, have been shown to have subtle and chronic effects on aquatic organisms. A number of studies have shown that levels of pharmaceuticals and personal care products ranging from ng/L to µg/L in wastewater treatment plant (WWTP) effluents (Miege et al., 2008; Clara et al., 2004; Joss et al., 2007). Pharmaceuticals and personal care products designed to have some biological effect even at low concentration and are concerned to cause the problems such as ecologically adverse effect or the occurrence of drug resistance bacteria in the aquatic environment (Okuda et al., 2008). Some of PPCPs are persistent, bioaccumulative, and/or harmful even if present at very low levels. There are no data on acute effects of PPCPs on human health, but the cumulative effect of long term exposure to a myriad of chemicals present

in the environment may have an adverse influence on human health and aquatic organisms. Thus, we need to begin to consider pharmaceuticals and personal care products as a source of water pollution. More research is necessary in order to understand what happens to PPCPs when we use them and how their presence in water systems may be affecting human health and aquatic populations. Regulation is necessary to limit the concentrations of these harmful compounds in effluents from wastewater treatment plants; the regulations need to be updated as more harmful constituent compounds are identified.

There is an increasing interest in the fate and behavior of these compounds within such facilities with the ultimate aim of optimizing treatment parameters to achieve the highest possible removal rates (Jones et al., 2006). While many PPCPs break down relatively quickly in effluents from WWTPs, many others are highly persistent to degradation. Therefore, the elimination of these persistent compounds is of elementary interest. It is important whether a relationship exists between achievable removal efficiencies and applied design criteria (Clara et al., 2005a).

The basic design and operating parameter used for WWTPs design is the sludge retention time (SRT). The SRT represents the average period of time during which the sludge has remained in the system. The SRT is the most critical parameter for activated sludge design as the SRT affects the treatment process performance, aeration tank volume and sludge production.

The objective of this thesis is to study the occurrence of PPCPs and to investigate a correlation between removal efficiency of PPCPs and the sludge retention time (SRT)

in the activated sludge process. The solid data from the literature was collected to allow comparison and evaluation of the removal efficiency with increasing SRTs in wastewater treatment and finally to reach qualitative conclusions.

BACKGROUND

Sludge Retention Time (SRT)

The SRT is the average amount of time the sludge spends in the aeration basin. The SRT is the reciprocal of the net specific growth rate (Eq. (1)). The maximum growth rate depends on temperature. Therefore, the SRT is also temperature dependent. Regarding the influence of μ_{max} on the SRT, a comparable dependency of the SRT on temperature can be assumed. For comparing the removal rates at the different treatment plants, total sludge retention time related to 20 °C ($SRT_{20°C}$) is calculated for all plants (Eq. (2)). This calculation is based on the SRT resulting from COD mass balance, the temperature (T) in the bioreactor and a correction coefficient ($f_p = 1.072$) for the temperature (Clara et al., 2004).

SRT = (active biomass in the system)/ (production rate of active biomass)

$$= \mu^{-1} \quad (1)$$

$$SRT_{20°C} = SRT_T \times 1.072^{(T - 20)} \quad (2)$$

Removal Mechanisms of Organic Compounds

The important removal pathways of organic compounds at wastewater treatment facilities are:

1. Volatilization
2. Adsorption to the sludge
3. Biodegradation/Biotransformation

Most researchers assume volatilization is negligible for many compounds because of the low values of the Henry coefficients (K_H) of compounds (Clara et al., 2005b; Radjenovic et al., 2006). This suggests that the PPCPs are being eliminated by biodegradation and sorption. Many pharmaceuticals are relatively hydrophilic and their sorption to sludge is limited by this hydrophilic nature and their with K_{ow} values (Jones et al., 2005). This limited sorption (low sorption coefficient or K_d) has led many researchers (Joss et al., 2006; Kimura et al., 2007; Wever et al., 2007) to conclude that the main mechanism of elimination of pharmaceuticals in the biological processes is biodegradation. However, there are always exceptions. For example, fluoroquinolones are very hydrophilic compounds, but adsorption to the sludge is the main elimination process in the wastewater treatment plants (Xu et al., 2007). Fluoroquinolone sorption may be favored by electrostatic interactions with the cell membranes of microorganisms. The removal of musk fragrances such as galaxolide and tonalide is mainly due to sorption onto sludge (Joss et al., 2005).

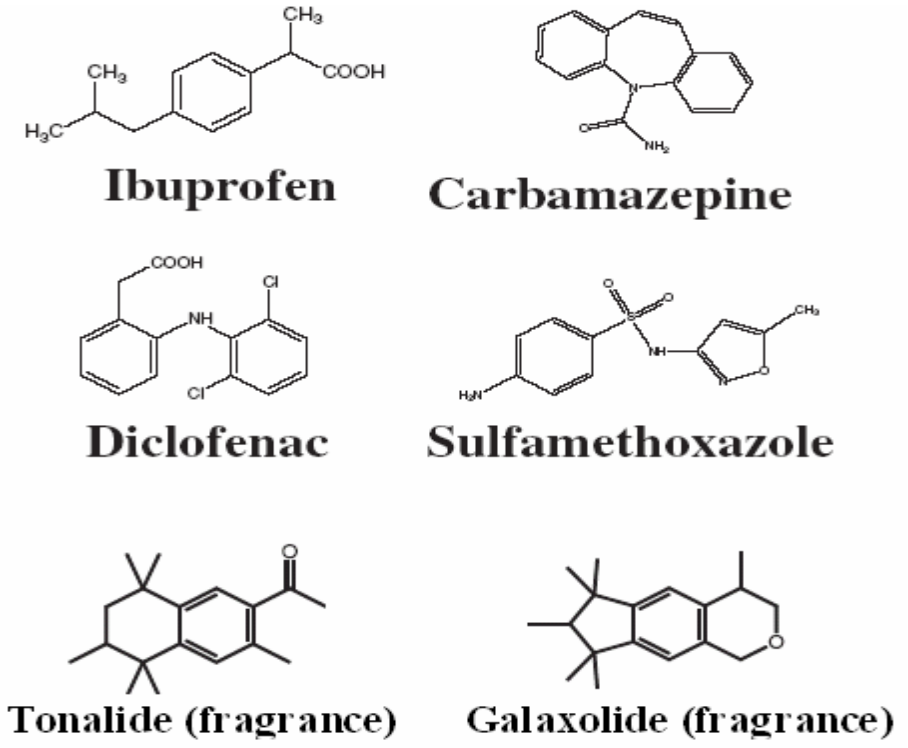
Characterization of Pharmaceuticals & Personal Care Products (PPCPs)

Pharmaceuticals are designed with the intention of performing a biological effect. For this reason, the compounds often have similar physical and chemical behavior. They are often lipophilic, in order to be able to pass through membranes, and are persistent to avoid inactivation before having their intended effects. Thus, many of the compounds have the necessary properties to bioaccumulate and provoke effects in aquatic or terrestrial ecosystems (Halling – Sorensen et al., 1998). Figure 1 shows common pharmaceuticals and personal care products found in WWTPs. Pharmaceutical molecules

often have many functional groups, such as carboxylic acids, aldehydes and amines, which make the binding capacities of the molecules to solids dependent on pH or other constituents in the solid matrix.

A number of compounds commonly used in a variety of personal care products have been found in effluents of WWTPs. Often times, they are persistent, not biodegraded under the conventional processes in WWTPs nor through natural attenuation, and therefore they accumulate in natural waters and sediments. Thus, not only do these chemicals enter water systems in increasing amounts through human use and insufficient wastewater treatment of personal care products, but can also accumulate in water systems or in organisms. For example, fragrances are hydrophobic and tend to resist biodegradation, they can be found accumulated in aquatic organisms and sediments (Schwarzbauzer et al., 2006).

Figure. 1. Common compounds used in PPCPs



Classification of PPCPs

Table 1 shows a classification of major pharmaceuticals and personal care products of different therapeutic classes (Miege et al., 2008).

Table. 1. PPCPs commonly found in WWTPs

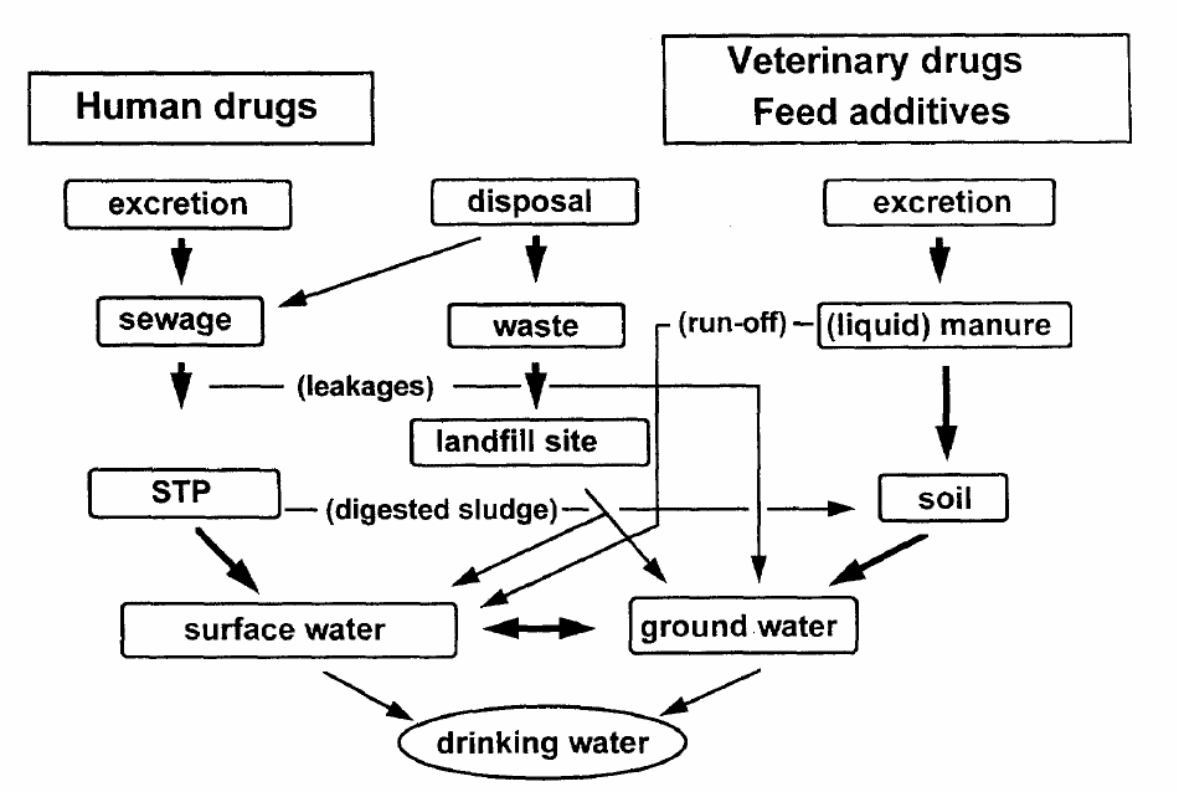
Therapeutic Class	Molecules
Analgesics and anti-inflammatory	Ibuprofen, Naproxen, Diclofenac, Ketoprofen, Mefenamic acid
Antibiotic	Sulfamethoxazole, Ciprofloxacin, Roxithromycin, Erythromycin, Norfloxacin
Antiepileptics	Carbamazepine, Diazepam
Beta – blocker	Atenolo, Metoprolo, Propanolol
Disinfectant	Triclosan
Hormone	Estrone, Estriole, 17 β-estradiol
Lipid regulator	Bezafibrate, Gemfibrozil
Metabolite	Clofibric acid
Personal care product	Galaxolide, Tonalide

Sources of Pharmaceuticals

Figure 2 shows the sources and pathways of pharmaceuticals. Due to their low volatility, pharmaceuticals can be introduced into the environment via municipal treatment discharge following human use through improper industrial/retail disposal methods and via runoff or spread of sludge from agricultural use in livestock production. In addition, a large amount of pharmaceuticals is being used annually. They are mainly excreted in urine or feces. The level of pharmaceuticals is mainly in the range of ng/L or some cases to µg/L. In short, the fate of pharmaceuticals is varied and strongly depends on certain conditions of environment. They are degraded by biodegradation or sorption into sediment. Although a large portion is degraded, a tiny amount of these substances

and their metabolites still exist in water bodies. By various routes, consequently, they reach surface water and groundwater and potentially go through to drinking water.

Fig. 2. Scheme of main fates of pharmaceuticals in environment (Ternes et al., 1998)



Methods for Analysis of PPCPs

Most researchers used LC-MS/MS (liquid chromatography with tandem mass spectrometry) and capillary column GC/MS (gas chromatography with mass spectrometry) for analysis of PPCPs. There are advantages and disadvantages of GC/MS and LC/MS methods depending upon the properties of the analytes. From the literature, we can conclude that GC is preferred for high vapor pressure, low molecular weight, non polar compounds, and can have limits of detection (LOD) in the low ng/L concentrations. Unfortunately GC analysis is time consuming and has variable recovery, which makes it too difficult and expensive for routine monitoring. LC/MS is preferable for polar and high molecular weight compounds. It can also obtain low ng/L LOD. Nevertheless, highly polar compounds are frequently difficult to analyze and concentration steps (extractions) are needed. Also the medium of the sample can produce matrix problems.

RESULTS AND DISCUSSION

Removal efficiencies of pharmaceuticals and personal care products for activated sludge processes

A literature survey of reported concentrations of PPCPs in activated sludge process effluents was performed and different removal efficiencies were noted. The most abundant compounds, their therapeutic classes and frequency of detection are shown in Table 2. The actual number of detections is larger than the numbers indicated because multiple locations within each reference are counted only once. Clara et al. (2004), Kreuzinger et al. (2004) Vieno et al. (2007), and Hashimoto et al. (2007) reported mean values of multiple observations. Miege et al. (2008) complied the largest database, surveying 113 separate publications, reporting therapeutic classes and frequency of detection. Full data for all PPCPs investigated are given in Appendix C. In order to limit the variability in reported removal efficiencies, references were restricted to those that reported the following information:

1. SRT and temperature;
2. Concentrations collected using 24 h composite sample;
3. Pilot and full scale WWTPs; and
4. Individual and mean removal rates.

For several compounds, such as acebutolol, sotanol, or aspirin, there is only one data set available; however, as mentioned above, it is a mean value of many measurements in multiple locations of WWTPs. Thus, these results can be considered reliable.

Table. 2. The abundant compounds and frequency in WWTPs

PPCPs	Frequency
Anti-inflammatory and analgesic	
Ibuprofen	9
Diclofenac	6
Fragrances	
Tonalide	4
Galaxolide	5
Antiepileptic	
Carbamazepine	7
Antibiotic	
Roxithromycin	5
Lipid regulator	
Bezafibrate	4
Contrast media	
Iopromide	5

High removal rates were observed with increasing SRT, and the trend was most obvious for ibuprofen, bisphenol – A and estrogens, with many observations (see Appendix C for compounds not included in Table 2). A possible explanation for the high removal rates of ibuprofen is elimination in the form of metabolization of hydroxyl-ibuprofen and carboxyl-ibuprofen (Strenn et al., 2004; Clara et al., 2004). In contrast, the low elimination rate and even the increase in concentration were observed for diclofenac and carbamazepine.

The K_d, K_{bio} and K_{ow} values of abundant compounds found in WWTPs are given in Table 3. Joss et al. (2006) created categories of removals to group rates of removals,

denoting that there are no distinct divisions of compound removal rates. Ibuprofen has a high K_{bio} which is consistent with the numerous observations (Joss et al., 2005; Jones et al., 2006; Nakada et al., 2006; Clara et al., 2004) of its efficient removal. The elimination of carbamazepine and diclofenac is negligible during wastewater treatment due to poor rate of biodegradation and negligible sorption. Even increases in concentrations of carbamazepine after treatment have been observed with the SRT of greater than 10d (Kreuzinger et al., 2004; Clara et al., 2004; Strenn et al., 2004). The most probable explanation for this is conversion of carbamazepine glucuronides and other conjugated metabolites to the parent compound by enzymatic processes in the treatment plant (Vieno et al., 2007). The removal of musk fragrances, galaxolide and tonalide, is expected due to their sorption to the sludge, but not biodegradation. Joss et al. (2005) also summarized in his review that two fragrances (galaxolide and tonalide) were mainly removed by sorption onto sludge.

Table. 3. Sorption coefficient, K_d, degradation rate constant, K_{bio}, and octanol-water partition coefficient, K_{ow} in WWTPs (Joss et al., 2006)

Compound	**K_d, L/g**	**K_{bio} for CAS, L/g/d**	**log K_{ow}**
Ibuprofen	0.007	21-35	3.97[c]
Naproxen	0.013	1.0-1.9	3.18[a]
Iopromide	0.011	1.6-2.5	-2.05[a]
Diclofenac	0.016	<0.1	1.13[d]
Carbamazepine	0.001[a]	0.008	2.45[b]
Galaxolide	5.2[a]	0.06	5.9[a]
Tonalide	10.8[a]	0.03	4.6[a]

	Removal mechanism			
		Minor	Partial	Major
	K_d, L/g	<0.3	0.3 - 1	> 1
	K_{bio} for CAS, L/g/d	< 0.1	0.1 - 10	> 10
		Strongly hydrophilic	Moderate	Strongly Hydrophobic
	Log K_{ow}	< 0	0 - 3	> 3

[a]: Joss et al., 2005
[b]: Vieno et al., 2007
[c]: Jones et al., 2005
[d]: The merck index, 2006

Influence of the sludge retention time (SRT) on the removal efficiency

By definition, the SRT is the mean residence time of the biomass in the system, and is functionally related to the growth rate of microorganisms. The collected removal rates for several abundant compounds in the activated sludge process are illustrated in Figure 3. High SRT is associated with better removal efficiency for most compounds except highly persistent compounds (carbamazepine, roxithromycin). The results of the removal rates for all the observed substances are qualitatively summarized in Appendix C. To determine a close correlation of removal of PPCPs to the SRT, compounds with high and steady removal were carefully chosen. In addition, to compare the results of the different sampling studies, the reported SRTs were all converted to 20°C using equation 2. The calculated removal efficiencies are shown in Figure 4. A clear dependency of removal efficiency on the SRT is easily observed for ibuprofen. At SRTs less than 1 day, no removal of ibuprofen is observed. Removal rates of more than 80% were observed at $SRT_{20°C}$ higher than 5 days. Also, Oppenheimer et al. (2007) had sufficient data to conclude that an SRT of 4.5 days is needed to remove 80% or more of ibuprofen. The term SRT_{80} is defined as the minimum SRT needed to consistently achieve 80% removal of the compound. These results confirm the high biodegradability of ibuprofen.

The results of the lipid regulator bezafibrate also show a significant dependency on the sludge retention time but with greater variation. Kreuzinger et al. (2004) gives no plausible reason for this variation. However, this variation could have been the result of uncontrolled variability in process conditions (Radjenovic et al., 2006).

It was expected that the musk fragrance tonalide would be mainly removed by sorption to sludge during wastewater treatment. Better removal efficiencies of tonalide were observed in the activated sludge process with longer SRTs; however, the calculated biological removal efficiencies at $SRT_{20°C}$ vary strongly in the middle range of the SRTs. No reasonable explanation could be found for the observed variations of removal for tonalide (Joss et al., 2005). The possible reason for great fluctuations is due to much more sensitivity to changes in operation conditions such as hydraulic retention time (HRT) or flow rate.

The antiepileptic drug carbamazepine is not well removed regardless of the SRTs due to its poor rate of biodegradation as well as its negligible sorption. In fact even higher concentrations were frequently found during wastewater treatment (Clara et al., 2005b; Vieno et al., 2007; Clara et al., 2004; Kreuzinger et al., 2004). Clara et al. (2004) observed almost twice as high concentrations of carbamazepine in the effluent at $SRT_{10°C}$ higher than 19 days. Also rates of removal of carbamazepine are strongly variable in the activated sludge process. No trend suggesting improved removal with increasing SRT was observed for diclofenac. For reference, Okuda et al. (2008) showed that ozonation process followed by biological treatment could significantly decrease PPCPs investigated including persistent compounds.

Although enhanced removal is not observed for all PPCPs investigated with increasing the SRT and no plausible explanation is given for the fluctuations in the observed removal of several substances, it is observed that the biological degradation of the PPCPs was higher with increasing the SRT. This is also valid if the substance is

degraded only as co-substrate, because the SRT necessary for the degradation of the primary substrate is the relevant parameter (Kreuzinger et al., 2004).

Figure. 3. Mean removal efficiency (%) for abundant compounds collected in WWTPs sorted by SRTs.

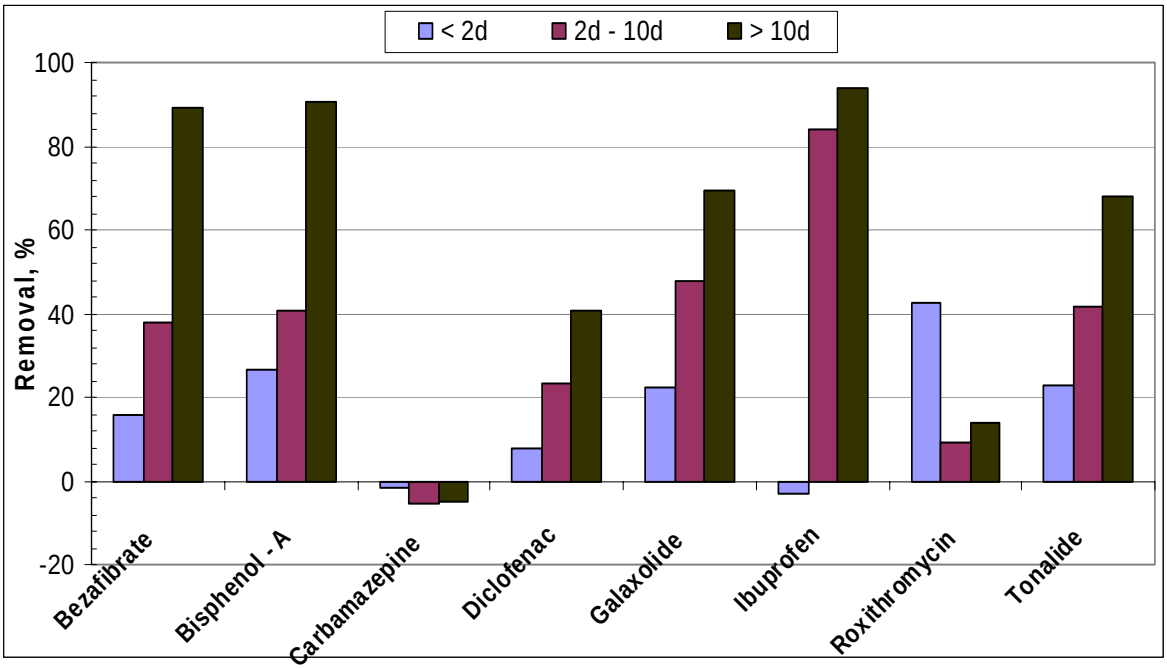
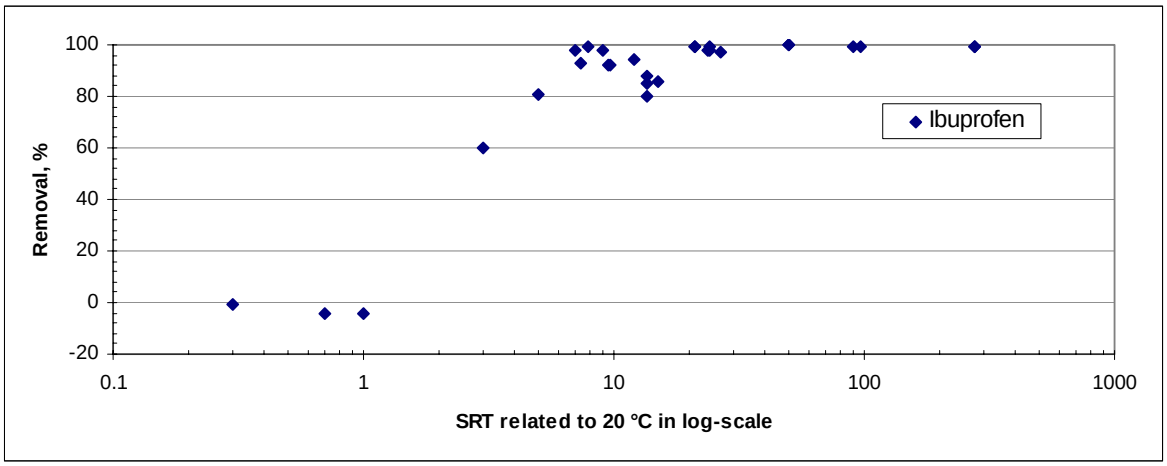
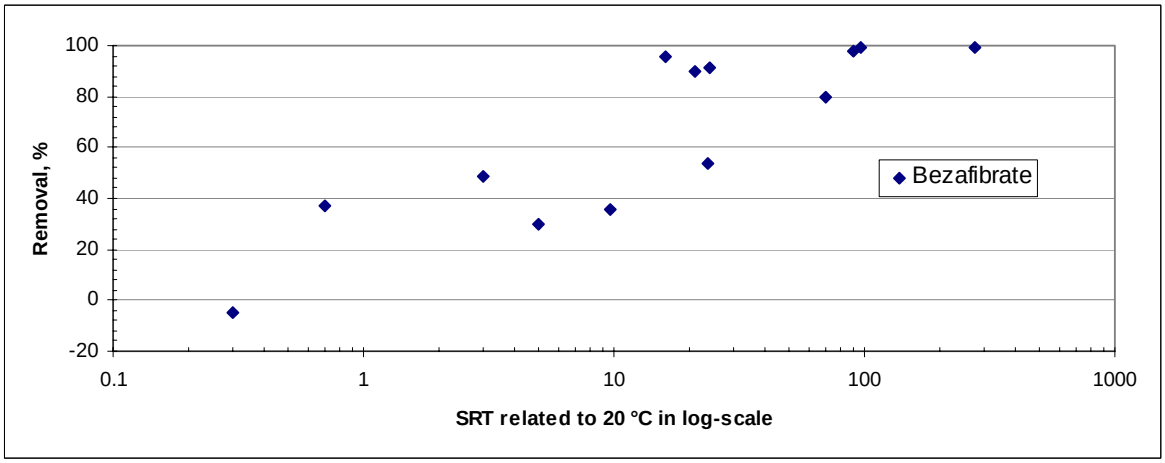
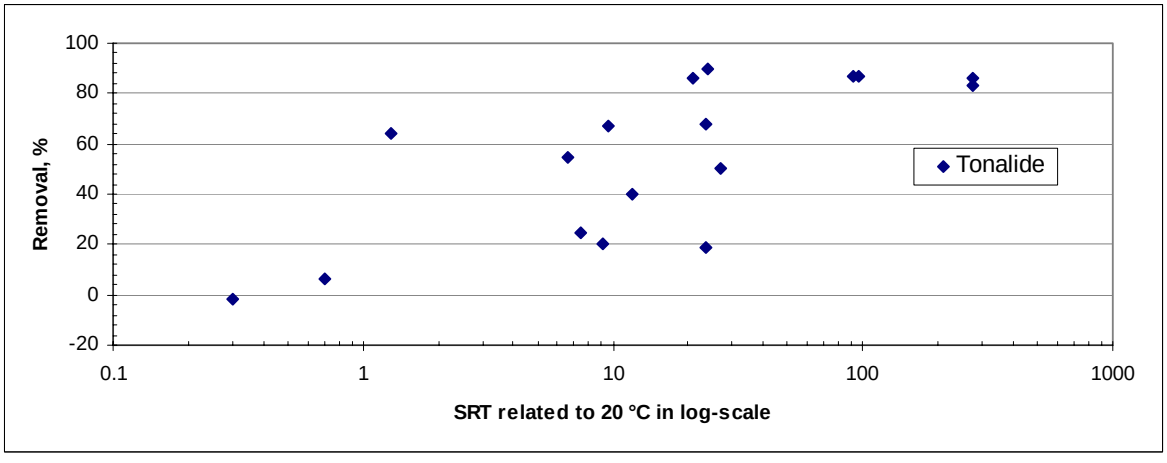


Figure. 4. Calculated removal efficiencies in relation to the $SRT_{20°C}$ for ibuprofen, bezafibrate and tonalide in the activated sludge process

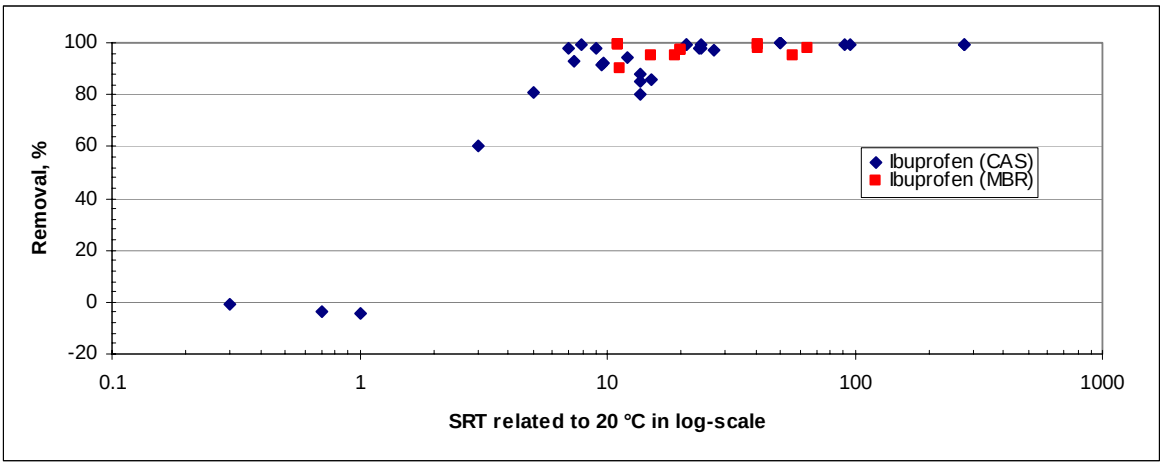


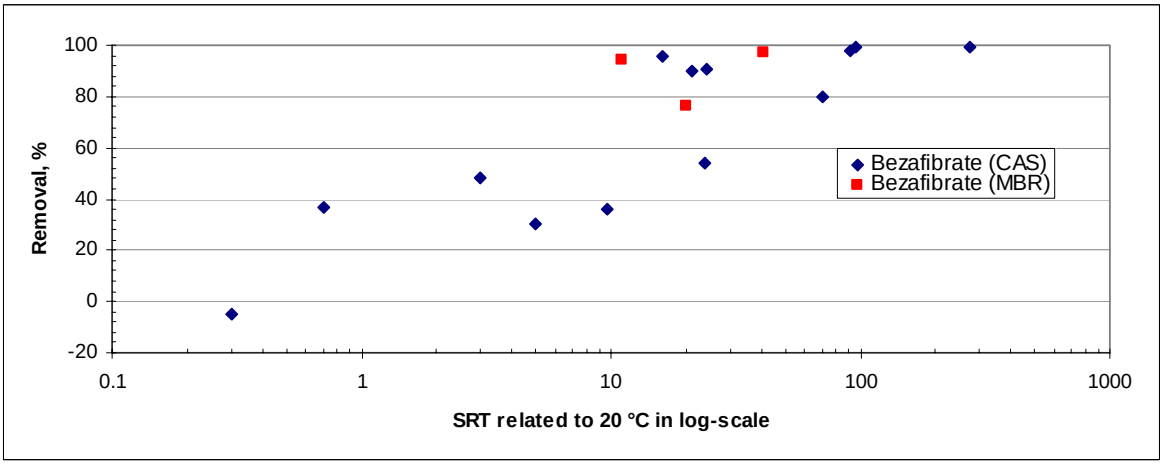


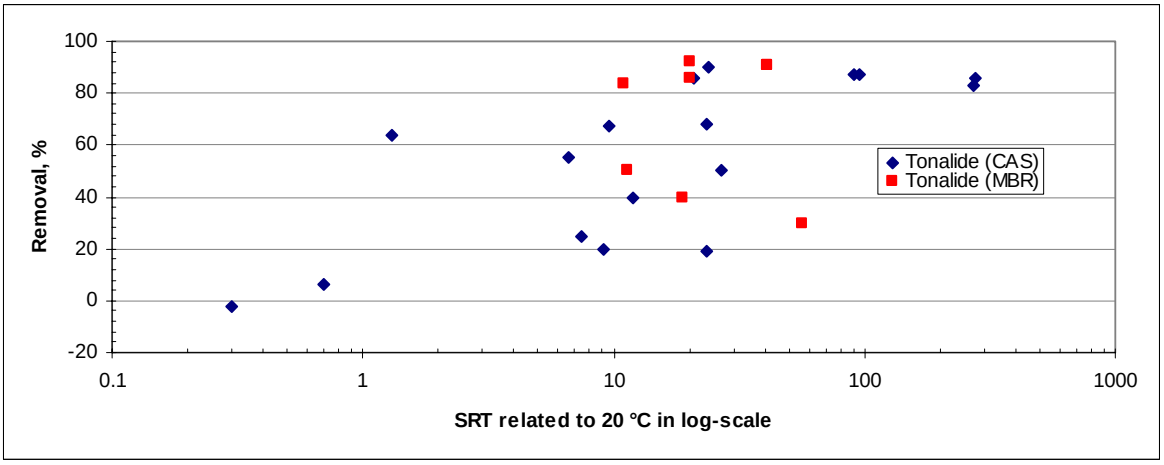


The influence of the treatment systems (MBR and CAS) on the removal efficiency of PPCPs has been argued (Kimura et al., 2007; Clara et al., 2004; Joss et al., 2005; Weaver et al., 2007). Some researchers reported improved removal efficiency in MBRs compared to CAS for several pharmaceuticals, ketoprofen and diclofenac (Kimura et al., 2005) and for 1,6- and 2,7 naphthalene disulfonate (Wever et al., 2007). However, Wever et al. (2007) observed no better removal of diclofenac in either the MBR or the CAS. Size exclusion, the prominent mechanism of MBR, does not affect removal of micropollutants since the molecular size is at least 100 times less than the pore size of the membranes. Clara et al. (2005b) also verified that ultrafiltration membranes do not allow any additional detention of PPCPs due to size sieving. An explanation for why MBRs seem to provide higher removal efficiency of micropollutants is that MBR is operated with longer SRTs than CAS. As mentioned earlier, elimination of PPCPs was mainly attributed to biodegradation due to their chemical properties. Longer SRTs allow for the slow growing bacteria to be retained, and eventually the bacterial population may become enriched to enhance the elimination of PPCPs. Clara et al. (2005a) investigated the treatment efficiency between CAS and MBR at comparable SRT, and the end result was that MBR operated with a comparable SRT showed no significant differences in the treatment efficiency with CAS. Therefore, the results of the author's study lead to the conclusion that sludge retention time (SRT) is the important parameter regarding to elimination of PPCPs. Figure 5 also supports the conclusion that MBRs are generally operated with longer SRTs and showed no better performance than did CASs.

Figure. 5. Calculated removal efficiencies in relation to the $SRT_{20°C}$ for ibuprofen, bezafibrate and tonalide in the CAS and MBR







CONCLUSION

Using the data from the literature, the following conclusions are made:

1. Literature observations consistently show that higher removal efficiencies are obtained for biodegradable pharmaceuticals and personal care products (PPCPs) in activated sludge plants operating and higher sludge retention times (SRTs);
2. Substances such as ibuprofen, bisphenol-A, and bezafibrate, showed a strong correlation between the removal rates and the SRT, whereas the antiepileptic drug, carbamazepine, was the most persistent substance and not affected by the SRT;
3. The main mechanism of elimination of most PPCPs is biodegradation due to low sorption rates and volatilization rates, whereas musk fragrances, tonalide and galaxolide, are well removed by sorption onto sludge;
4. High removal rates of PPCPs have been reported for membrane bioreactors (MBRs), but the observations noted in this thesis suggest that the improved removals are associated with higher SRTs, and not the process configuration. Most MBRs are operated at high SRT, whereas many activated sludge plants are operated at low SRT.

APPENDIX A: PROPERTIES OF COMPOUNDS

Name	Formula	CAS	$LogK_{ow}$	Solubility in water (mg/ml)	Usage	Types
Acebutolol	$C_{18}H_{28}N_2O_4$	37517-30-9	1.71[d]	0.3[d]	Beta blocker	Pharmaceuticals
Acetaminophen	$C_8H_9NO_2$	103-90-2	0.46[c]	very slightly sol in cold water	Tylenol & paradol	Pharmaceuticals
Aspirin	$C_9H_8O_4$	50-78-2		3.3 at 25°C	Analgesic	Pharmaceuticals
Atenolol	$C_{14}H_{22}N_2O_3$	29122-68-7	0.16[d]	slightly sol in water (13.3[d])	Antihypertensive	Pharmaceuticals
Benzophenone	$C_{13}H_{10}O$	119-61-9		insol in water	Sunscreen preparations	Personal care products
Benzyl Salicylate	$C_{14}H_{12}O_3$	118-58-1		slightly sol in water	Sunscreen preparations	Personal care products
Bezafibrate	$C_{19}H_{20}ClNO_4$	41859-67-0			LDL cholesterol control	Pharmaceuticals
Bisphenol-A	$C_{15}H_{16}O_2$	80-05-7		0.12-0.3 sol in alcohol, acetone	Estrogen receptor agonist	EDC
Butylated hydroxyanisole	$C_{11}H_{16}O_2$	25013-16-5		insol in water sol in fats,oils	Antioxidant	Personal care products
Butylated hydroxytoluene	$C_{15}H_{24}O$	128-37-0		insol in water sol in methanol,ethanol	Antioxidant, food additive as well as in cosmetics, pharmaceuticals	Personal care products pharmaceuticals
Butylbenzyl phthalate	$C_{19}H_{20}O_4$	85-68-7[a]	4.78,4.91[a]	2900 ± 1200 in DI water[a]	plasticizer	Plasticizer
Caffeine	$C_8H_{10}N_4O_2$	58-08-2	-0.07	22 at 25°C, 180 at 80°C	psychoactive stimulant	Pharmaceuticals
Carbamazepine	$C_{15}H_{12}N_2O$	298-46-4	2.45[d]	insol in water sol in alcohol,acetone	Anticonvulsant In treatment of pain w/ trigeminal neuralgia	Pharmaceuticals
Carisoprodol	$C_{12}H_{24}N_2O_4$	78-44-4		very sparingly sol in water 0.3 at 25°C, 1.4 at 50°C	Skeletal muscle relaxant	Pharmaceuticals
Chloramphenicol	$C_{11}H_{12}Cl_2N_2O_5$	56-75-7		2.5	Antibacterial use in eye drops & ointment	Pharmaceuticals
Ciprofloxacin	$C_{17}H_{18}FN_3O_3$	85721-33-1	0.28[d]	30[d]	Antilipemic	Pharmaceuticals
Clofibric acid	$C_{10}H_{11}ClO_3$	882-09-7			Blood lipid metabolite	Inhibitor
Crotamiton	$C_{13}H_{17}NO$	483-63-6			Antipruitic	Pharmaceuticals
Diazepam	$C_{16}H_{13}ClN_2O$	439-14-5		insol in water	Muscle relaxant	Pharmaceuticals
Diclofenac	$C_{14}H_{11}Cl_2NO_2$	15307-86-5	1.13	DI water>9 methanol>24,acetone>6	Anti-inflammatory & analgesic	Pharmaceuticals

Diethyltoluamide	$C_{12}H_{17}NO$	134-62-3	2.02	9.9 at 25°C	Insect repellent	Inhibitor
Erythromycin	$C_{37}H_{67}NO_{13}$	114-07-8		2 sol in alcohol,acetone	Antibacterial	Pharmaceuticals
Estradiole	$C_{18}H_{24}O_2$	50-28-2		insol in water sol in alcohol,acetone	The major estrogen in humans	EDC
Estriol	$C_{18}H_{24}O_3$	50-27-1		insol in water sol in alcohol,chloroform	One of the three main estrogens	EDC
Estrone	$C_{18}H_{22}O_2$	53-16-7		0.03 sol in dioxane, veg oils	The least prevalent of the three hormone	EDC
Fenofibrate	$C_{20}H_{21}ClO_4$	49562-28-9		insol in water	Blood lipid regulator	Pharmaceuticals
Fenoprofen	$C_{15}H_{14}O_3$	31879-05-7		2.5 at 37°C	Analgesic	Pharmaceuticals
Galaxolide	$C_{18}H_{26}O$	1222-05-5[b]	5.9[b]		Fragrance	Personal care products
Gemfibrozil	$C_{15}H_{22}O_3$	25812-30-0			Blood lipid regulator	Pharmaceuticals
Hydrochlorothiazide	$C_7H_8ClN_3O_4S_2$	58-93-5		insol in water	Antibiotic	Pharmaceuticals
Ibuprofen	$C_{13}H_{18}O_2$	15687-27-1	3.97[c]	relatively insol (0.021) in water	Anti-inflammatory drug (Advil)	Pharmaceuticals
Indomethacin	$C_{19}H_{16}ClNO_4$	53-86-1		insol in water	Anti-inflammatory & analgesic	Pharmaceuticals
Iopromide	$C_{18}H2_4I_3N_3O_8$	73334-07-3	-2.05[b]		Diagnostic aid	Pharmaceuticals
Ketoprofen	$C_{16}H_{14}O_3$	22071-15-4		slightly sol in water	Analgesic	Pharmaceuticals
Mefenamic acid	$C_{15}H_{15}NO_2$	61-68-7	5.12[c]	0.01 at pH 7.1	Pain reliever as Ponstel	Pharmaceuticals
Methylparaben	$C_8H_8O_3$	99-76-3		0.04	Preservative	Personal care products
Metoprolol	$C_{15}H_{25}NO_3$	37350-58-6	1.69[d]	4.78[d]	Antihypertensive	Pharmaceuticals
Naproxen	$C_{14}H_{14}O_3$	22204-53-1	3.18[b]	insol in water	Pain reliever	Pharmaceuticals
Nonylphenol	$C_{15}H_{24}O$	25154-52-3	3.3 at 20C[a]	insol in water	Plasticizers,oil additives	EDC, plasticizer
Norfloxacin	$C_{16}H_{18}FN_3O_3$	70458-96-7	-0.34	0.28 at 25°C in water solubility in water is pH dependent	Antibacterial	Pharmaceuticals
Octymethoxycinnamate	$C_{18}H_{26}O_3$	5466-77-3			UV screen	Personal care products
Ofloxacin	$C_{18}H_{20}FN_3O_4$	82419-36-1	-0.39[d]	sparingly sol in water	Anthrax(Floxin)	Pharmaceuticals
Oxybenzone	$C_{14}H_{12}O_3$	131-57-7		readily sol in most org solvents	UV light absorber & stabilizer	Personal care products

Paroxetine	$C_{19}H_{20}FNO_3$	61869-08-7			Psychiatric	Pharmaceuticals
Propranolol HCl	$C_{16}H_{21}NO_2HCl$	318-98-9	0.74[c]	3.01	Antihypertensive	Pharmaceuticals
Propyphenazone	$C_{14}H_{18}N_2O$	479-92-5		2.4 at 16.5°C	Analgesic	Pharmaceuticals
Ranitidine	$C_{13}H_{22}N_4O_3S$	66357-35-5			Anti-ulcer agents	Pharmaceuticals
Roxithromycin	$C_{41}H_{76}N_2O_{15}$	80214-83-1	2.75[b]		Antibiotic	Pharmaceuticals
Salbutamol	$C_{13}H_{21}NO_3$	18559-94-9	0.64[c]		Adrenergic receptor	Pharmaceuticals
Sotalol	$C_{12}H_{20}N_2O_3S$	3930-20-9	0.24[d]	137[d]	Antihypertensive	Pharmaceuticals
Sulfadiazine	$C_{10}H_{10}N_4O_2S$	68-35-9		sparingly sol in water at 37°C 0.013 at pH 5.5, 0.02 at pH 7.5	Antibiotic	Pharmaceuticals
Sulfadimidine	$C_{12}H_{14}N_4O_2S$	57-68-1		sol in water at 37°C, 1.92 at pH7, increase w/ pH	Antibacterial	Pharmaceuticals
Sulfamethoxazole	$C_{10}H_{11}N_3O_3S$	723-46-6	0.89[b]		Antibacterial agent for E-coli	Pharmaceuticals
Tetracycline	$C_{22}H_{24}N_2O_8$	60-54-8		1.7 at 28°C in water	Antibacterial	Pharmaceuticals
Thymol	$C_{10}H_{14}O$	89-83-8	3.3[a]	1 volatilizes in water vapors	Antiseptic	Pharmaceuticals
Tonalide	$C_{18}H_{26}O$	21145-77-7[b]	4.6[b]		Fragrance	Personal care products
Triclosan	$C_{12}H_7Cl_3O_2$	3380-34-5		insol in water	Preservative in foods cosmetics	Personal care products
Trimethoprim	$C_{14}H_{18}N_4O_3$	738-70-5	0.91[e]	10 at 25°C in water	Antibacterial	Pharmaceuticals
Triphenylphosphate	$C_{18}H_{15}O_4P$	115-86-6	4.59[a]	insol in water	Plasticizer	Plasticizer
Tris phosphine	$C_9H_{16}O_6PCl$	51805-45-9			Antioxidant	Personal care products

Data from the merck index, 2006
[a]: Handbook of environmental data on organic chemicals, 2001
[b]: Joss et al., 2005
[c]: Jones et al., 2005
[d]: Vieno et al., 2007
[e]: Batt et al., 2006

APPENDIX B: CHARACTERIZATION OF THE TREATMENT PROCESSES FOR THE DATA SET

Ref	Author	Location	Plant (Type, SRT)	Size, HRT,PE (*10^3)	Temp, °C
1	Joss et al (2005)	Swiss	WWTP1 - CAS w/MBR, SRT(10d~12d)/CAS, SRT(16d, 33d, 75d)/MBR WWTP2 - CAS w/FBR, SRT(22d~24d)/CAS	HRT: CAS(7.3h), MBR(13h), PE(55) HRT: CAS(16.8h), FBR(0.7h), PE(80)	CAS1: 13~16, MBR: 12~16 CAS2: 12~21, FBR: 12~19
2	Jones et al (2006)	South England	1 STP - CAS SRT(13d)	HRT(13.5h) PE(150)	20.6
3	Xu et al (2007)	South China	4 STPs 1:CAS, SRT(4.5h-6h) 2:Oxidation ditch, SRT(NA) 3:CAS, SRT(5.6h-8h) 4:Chem enhanced,SRT (3h-4h)	1: HRT(22h), PE(80) 2:HRT(12h-18h),PE(1,050) 3:HRT(15h-22h),PE(300) 4:HRT(8h-12h),PE(3,500)	
4	Oppenheimer et al (2007)	U.S.	6 facilities, SRT ranging from 0.5d to 30d	Size(MGD): 5-300	
5	Kreuzinger et al (2004)	Austria	4 WWTPs 1: CAS(1stage), SRT(24d,96d,275d) 2 CAS(1 stage),SRT(0.7d) 3:CAS(1stage),SRT(23.6d) 4:CAS(2 stages),SRT(0.3, 9.6d)	1: PE(7), 2:PE(2,500) 3:PE(135), 4:PE(167)	set at T =20
6	Clara et al (2004)	Austria	5 WWTPs 1: CAS, SRT(2d) 2:CAS(2 stages + anaerobic sludge),SRT(19d) 3:CAS+anaerobic sludge, SRT(48d) 4:CAS,SRT(100d/100d/42d), 5:MBR,SRT(22d/82d/40d)	1: PE(2,500), 2:PE(167) 3:PE(135), 4:PE(6), 5:PE(0.05)	set at T =10
7	Nakada (2006)	Tokyo, Japan	5 STPs w/ CAS SRT(d): 3.8, 4.6, 5.8, 5.0, 8.4	Size(MGD): 170,108,85,317,55 HRT(h): 8.6,8.0,9.4,7.1,8.9 PE: 709,731,764,2020,464	Samples collected seasonally
8	Vieno et al (2007)	Finland	12 STPs w/ CAS mostly SRT(d): 2 to 20 variously	Size(MGD): 0.2 - 62 HRT(h): 1.5 - 20 PE: 2.4 - 740	
9	Hashimoto et al (2007)	Japan	10 WWTPs w/ CAS SRT(d): 2-10	Size(MGD): 1.5 - 9.9 HRT(h): 6 - 26 PE: 16 - 131	Summer: 20 - 28 Winter: 13 - 25

10	Batt et al (2007)	U.S.	3 WWTPs (CAS) WWTP 1: SRT(d): (6 and 49 for stage 1 and 2, respectively) WWTP2: SRT(d): 15 WWTP3: SRT(d): 17	Size(MGD): 0.8,4.5 and 30 HRT(h): 1 - 4	
11	Batt et al (2006)		Batch experiment & one WWTP: stage1(CAS) w/SRT(6d) stage2(CAS w/ nitrification) w/ SRT(49d)	Size(MGD): 30 HRT(h):1,2	
12	Kimura et al (2007)	Japan	1 WWTP (SRT(d):7) 2 MBRs (SRT(d):15, 65)	Size(MGD): 1WWTP (33) 2 MBRs	Summer(Aug - Oct)
13	Radjenovic et al (2007)	Spain	Lab - scale MBR compared with removal in CAS SRT of MBR was set as infinite SRT of CAS w/ nitrification(d):3	CAS system w/ nitrification of WWTP Size(MGD): 5.8 HRT(h): 14	
14	Clara et al[b] (2004)	Austria	1 STP(CAS), SRT(d): 91, 275, 21 1 MBR SRT(d): 11, 41, 20	PE: 7	set at T = 20
15	Strenn et al (2004)	Austria	12 SWPs SRT(d): 1,4, 17, 29		set at T = 20
16	Clara et al (2005)	Austria	1 MBR: SRT(d): 10,27,55 3 WWTPs: WWTP1: SRT(d): 114, 237, 52 WWTP2: SRT(d): 2 WWTP3: SRT(d): 46		Samples collected seasonally

APPENDIX C: REMOVAL OF PPCPs IN RELATION TO THE SRTs IN THE DIFFERENT TRATMENT PROCESSES

	SRT Range				
	< 2d	**2d - 5d**	**5d -10d**	**10d -20d**	**> 20d**
Compound	**V. Low**	**Low**	**Medium**	**High**	**V. High**
3 - Phenylpropionate	< 1d for SRT80/Ref4				
Acebutolol		60[b]			
Acetaminophen				91.9/Ref2(CAS)	
Aspirin		> 90[a]			
Atenolol		63[b]			
Benzophenone				12d for SRT80/Ref4	
Benzyl Salicylate		4.5d for SRT80/Ref4			
Bezafibrate	-5,37 at 20°C/Ref5(CAS)	48.4 /Ref13(CAS w/ nit)	36 at 20°C/Ref5(CAS) 30 at 20°C/Ref15(CAS)	94,76 at 20°C/Ref5(MBR) 96 at 20°C/Ref15(CAS)	54,91,99,99 at 20°C/Ref5(CAS) 97 at 20°C/Ref5(MBR) 98,98,90 at 20°C/Ref14(CAS) 80 at 20°C/Ref15(CAS)
Bisphenol-A	47,23 at 20°C/Ref5(CAS)	10.5 at 10°C/Ref6(CAS) > 90[a]	39 at 20°C/Ref5(CAS)	42.4 at 10°C/Ref6(CAS) 98,97 at 20°C/Ref5(MBR)	95,97,87,68 at 20°C/Ref5(CAS) 99 at 20°C/Ref5(MBR) 83,99,99,97 at 10°C/Ref6(CAS) 99,99,93 at 10°C/Ref6(MBR)
Butylated hydroxyanisole			>7d for SRT80/Ref4		
Butylbenzyl Phthalate		4.5d for SRT80/Ref4			
Caffeine		4.5d for SRT80/Ref4			

Carbamazepine	0,-3 at 20°C/Ref5(CAS) 0 at 20°C/Ref15(CAS)	-3 at 10°C/Ref6(CAS) < 45[a] 2 at 20°C/Ref15(CAS)	-44[b] 35 at 20°C/Ref5(CAS)	20,-25,3 at 14,13,16°C/Ref1(CAS) 25,-20,-5 at 15,12,16°C/Ref1(MBR) 11,-8 at 20°C/Ref5(MBR) -67 at 10°C/Ref6(CAS) 11 at 20°C/Ref14(MBR) -9 at 20°C/Ref15(CAS)	10,-20 at 21,12°C/Ref1(CAS) 9 at 20°C/Ref5(MBR) 14,10 at 20°C/Ref5(CAS) 14,-11,-35 at 10°C/Ref6(CAS) 13,4,-13 at 10°C/Ref6(MBR) 14,-16,-42 at 20°C/Ref14(CAS) -7,-14 at 20°C/Ref14(MBR) 2 at 20°C/Ref15(CAS)
Chloramphenicol	45/Ref3(ox)				
Ciprofloxacin		86[b]	59/Ref10(amhest- CAS1)	71,64/Ref10	0/Ref10(Amherst CAS2 w/nit)
Clofibric acid		27.7/Ref13(CAS w/ nit)	50/Ref12	50/Ref12	82/Ref12
Crotamiton		25 (range 0-60%)[a]			
DEET		40 (range:10-95%)[a]		>15 for SRT80/Ref4	
Diazepam					25,20,23 at 20°C/Ref5(CAS)
Diclofenac	7.9 at 20°C/Ref5(CAS) 8 at 20°C/Ref15(CAS)	7.1 at 10°C/Ref6(CAS) 50.1/ Ref13(CAS w/ nit) 25 at 20°C/Ref15(CAS)	9 at 20°C/Ref5(CAS) 42/Ref12(CAS)	35,20,30 at 14,13,16°C/Ref1(CAS) 35,15,40 at 15,12,16°C/Ref1(MBR) -8,39 at 20°C/Ref5(MBR) -25 at 10°C/Ref6(CAS) 51/Ref12(MBR) -5 at 20°C/Ref14(MBR) 9 at 20°C/Ref15(CAS)	35,30 at 21,12°C/Ref1(CAS) 13,52,46,69 at 20°C/Ref5(CAS) 51 at 20°C/Ref5(MBR) 14,53,63,47 at 10°C/Ref6(CAS) -7,51,33 at 10°C/Ref6(MBR) 82/Ref12(MBR) 57,70,50 at 20°C/Ref14(CAS) 52,40 at 20°C/Ref14(MBR) 1 at 20°C/Ref15(CAS)
Erythromycin	45,15,45/ Ref3(CAS,CAS,chem)	23,8/ Ref13(CAS w/ nit)			
Estradiol		90%[a]	85.7[c]		
Estriol		18 at 10°C/Ref6(CAS) 100%[a]	99.5[c]	26 at 10°C/Ref6(CAS)	100,100,100,100 at 10°C/Ref6(CAS) 100,100,100 at 10°C/Ref6(MBR)
Estrone		-112 at 10°C/Ref6(CAS) 86[a]	-55.9[c]	84.3 at 10°C/Ref6(CAS)	100,94,100,98 at 10°C/Ref6(CAS) 97,28,100 at 10°C/Ref6(MBR)
Fenoprofen		85(range 65-95%)[a]			

Galaxolide	2,27 at 20°C/Ref5(CAS) 38 at 20°C/Ref16(CAS)		56 at 20°C/Ref5(CAS)	60,35,40 at 14,13,16°C/Ref1(CAS) 60,40,30 at 15,12,16°C/Ref1(MBR) 85,90 at 20°C/Ref5(MBR) 84 at 20°C/Ref14(MBR)	50,50 at 21,12°C/Ref1(CAS) >30d for SRT80/Ref4 92 at 20°C/Ref5(MBR) 44,85,86,89 at 20°C/Ref5(CAS) 90,83 at 20°C/Ref14(MBR) 85,86,81,36 at 20°C/Ref16(CAS)
Gemfibrozil		38.8/ Ref13(CAS w/ nit)			
Hydrochlorothiazide		76.3/ Ref13(CAS w/ nit)			
Ibuprofen	-1, -4 at 20°C/Ref5(CAS) -4.3 at 10°C/Ref6(CAS)	4.5d for SRT80/Ref4 over 90%[a] 82.5/ Ref13(CAS w/ nit) 60 at 20°C/Ref15(CAS)	92 at 20°C/Ref5(CAS) 98/Ref12(CAS) 81 at 20°C/Ref15(CAS)	99,93,98 at 14,13,16°C/Ref1(CAS1) 90,95,95 at 15,12,16°C/Ref1(MBR) 85,80,88,91/Ref2(CAS) 99 at 20°C/Ref5(MBR) 91.8 at 10°C/Ref6(CAS) 95/Ref12(MBR) 86 at 20°C/Ref15(CAS)	97,94 at 21,12°C/Ref1(CAS2) 97,99 at 20°C/Ref5(MBR) 98 at 10°C/Ref6(CAS) 100, 100, 99 at 10°C/Ref6(CAS) 99, 99, 97 at 10°C/Ref6(MBR) 98/Ref12(MBR) 99,99,99 at 20°C/Ref14(CAS)
Indomethacin		23.4/ Ref13(CAS w/ nit)			
Iopromide	-8, 0 at 20°C/Ref5(CAS) -32 at 20°C/Ref16(CAS)		50 at 20°C/Ref5(CAS) -22/Ref11(CAS)	45,30,80 at 14,13,16°C/Ref1(CAS) 40,65,75 at 15,12,16°C/Ref1(MBR)	92,60 at 21,12°C/Ref1(CAS) 25 at 20°C/Ref5(CAS) 61/Ref11(CAS w/ nit) -861 at 20°C/Ref16(CAS)
Ketoprofen		45(range: 10-70%)[a] 51.5/ Ref13(CAS w/ nit)	55/Ref12(CAS)	83/Ref12(MBR)	>98/Ref12(MBR)
Mefenamic acid		29.4/ Ref13(CAS w/ nit)	72/Ref12(CAS)	91.54/Ref2(CAS) 77/Ref12(MBR)	93/Ref12(MBR)
Methyl-3-phenylpropionate	< 1d for SRT80/Ref4				
Methylparaben	< 1d for SRT80/Ref4				
Metoprolol		34[b]			
Musk Ketone					>30d for SRT80/Ref4

Naproxen		45(range: 0-80%)[a] 85.1/ Ref13(CAS w/ nit)	64/Ref12(CAS)	75,80 at 13,16°C/Ref1(CAS) 75,80,77 at 15,12,16°C/Ref1(MBR) 96/Ref12(MBR)	65,70 at 21,12°C/Ref1(CAS) >96/Ref12(MBR)
Nonylphenol	81 at 20°C/Ref16(CAS)	70(range: 60-75%)[a]	5d for SRT80/Ref11	10d for SRT80/Ref11 91 at 20°C/Ref16(MBR)	88,90,90,78 at 20°C/Ref16(CAS) 89,85 at 20°C/Ref16(MBR)
Norfloxacin	80,65,50,65/ Ref3(CAS,ox,CAS,chem)	ND[b]			
Octylmethoxycinnamate		4.5d for SRT80/Ref4			
Octylphenol	87 at 20°C/Ref16(CAS)	< 45[a]		45 at 20°C/Ref16(MBR)	75,100,93,27 at 20°C/Ref16(CAS) 100,66 at 20°C/Ref16(MBR)
Ofloxacin	70,60,40,55/ Ref3(CAS,ox,CAS,chem)	83%[b] 23.8/Ref13(CAS w/ nit)			
Oxybenzone		4.5d for SRT80/Ref4			
Paroxetine		90.6/ Ref13(CAS w/ nit)			
Propyphenazone		negative%[a] 42.7/ Ref13(CAS w/ nit)			
Ranitidine		42.2/ Ref13(CAS w/ nit)			
Roxithromycin	65,55,75/ Ref3(CAS,CAS,chem) -8,27 at 20°C/Ref5(CAS)		-4 at 20°C/Ref5(CAS)	20,40,-20 at 14,13,16°C/Ref1(CAS) 40,60,55 at 15,12,16°C/Ref1(MBR) 75 at 20°C/Ref5(MBR) 100 at 20°C/Ref16(MBR)	40,5 at 21,12°C/Ref1(CAS) 58,61 at 20°C/Ref5(CAS) -58,44,41,-80 at 20°C/Ref16(CAS) 34,74 at 20°C/Ref16(MBR)
Salbutamol				94.6/Ref2(CAS)	
Sotalol		54[b]			
Sulfadiazine	50/Ref3(ox)				

Sulfadimidine	50,50/Ref3(CAS,ox)				
Sulfamethoxazole	35,64/Ref3(ox,chem) -279 at 20°C/Ref16(CAS)	55.6/Ref13(CAS w/ nit)	57/Ref10(Amhest-CAS1)	55,55 at 14,13°C/Ref1(CAS) 90,75,70 at 15,12,16°C/Ref1(MBR) 48,75/Ref10 57 at 20°C/Ref15(MBR) 61 at 20°C/Ref16(MBR)	70,65 at 21,12°C/Ref1(CAS) 33,62 at 20°C/Ref5(CAS) 42/Ref10(Amherst CAS2 w/ nit) 66, 32 at 20°C/Ref16(CAS)
Tetracycline			63/Ref10(Amhest- CAS1)	81,33/Ref10	59/Ref10(Amherst CAS2 w/ nit)
Thymol		95[a]			
Tonalide	-2,6 at 20°C/Ref5(CAS) 64 at 20°C/Ref16(CAS)		67 at 20°C/Ref5(CAS)	55,25,20 at 14,13,16°C/Ref1(CAS) 50,40,30 at 15,12,16°C/Ref1(MBR) 84 at 20°C/Ref14(MBR)	40,50 at 21,12°C/Ref1(CAS) 68,90,87,86 at 20°C/Ref5(CAS) 87,83,19 at 20°C/Ref16(CAS) 91,86 at 20°C/Ref16(MBR)
Total NP	22 at 20°C/Ref5(CAS)		57 at 20°C/Ref5(CAS)	80,88 at 20°C/Ref5(MBR)	80,91,91,28 at 20°C/Ref5(CAS) 69 at 20°C/Ref5(MBR)
Triclosan		70%(range:45-92%)[a]		10d for SRT80/Ref4	
Trimethoprim			-4/Ref10(Amherst CAS1) -1/Ref11(CAS)	97,83/Ref10	68/Ref10(Amherst- CAS2 w/ nit) 50/Ref11(CAS w/ nit)
Triphenylphosphate			>5d for SRT80/Ref4		

Ref 1: Joss et al., 2005	Ref 5: Kreuzinger et al., 2004	Ref 9: Hashimoto et al., 2007	Ref 13: Radjenovic et al., 2007
Ref 2: Jones et al., 2006	Ref 6: Clara et al., 2005	Ref 10: Batt et al., 2007	Ref 14: Clara et al., 2004
Ref 3: Xu et al, 2007	Ref 7: Nakada et al., 2006	Ref 11: Batt et al., 2006	Ref 15: Strenn et al., 2004
Ref 4: Oppenheimer et al., 2007	Ref 8: Vieno et al., 2007	Ref 12: Kimura et al., 2007	Ref 16: Clara et al., 2005

[a]: Average removal rate for SRT 3.8 to 8.4d (Ref7)
[b]: Average removal rate for SRT 2 to 20d (Ref8)
[C]: Average removal rate for SRT 2-10d (Ref9)
SRT80: The minimum SRT value needed to achieve compound removal greater than 80%
CAS: Conventional activated sludge
MBR: Membrane bioreactor
nit: Nitrification
ox: Oxidation ditch
chem: Chemically enhanced
ND: Not detected

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