Household chemicals and personal care products as sources for xenobiotic organic compounds in grey wastewater

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Abstract

Despite contributing 75% of the total wastewater flow to domestic sewers, little is currently known concerning the detailed production patterns and characteristics of grey wastewater. In this study, an inventory of the consumption of household chemicals including a diary survey of water-consuming activities was carried out over seven consecutive days in a block of flats. In total 290 parameters in 92 household chemicals were registered in the inventory in which 30 out of 38 tenants participated. The study was accompanied by quantitative analyses of selected parameters and a screening for organic components in grey wastewater. More than 190 individual components were identified by GC-MS. Identified substances were grouped into eight substance classes based on their application and their concentrations were semi-quantitatively assessed. Several fragrances like citronellol, hexyl cinnamic aldehyde and menthol as well as some preservatives, e.g. citric acid and triclosan, were identified. The measurements also showed that unwanted and unexpected compounds like drugs and pesticides could be present, as well as chemicals not directly deriving from household chemicals or personal care products, e.g. flame-retardants. The inventory provided detailed information about the consumption of various types of household chemicals, but no information on compound concentrations could be assessed due to the limited data in the list of contents of the household chemicals. It was shown that tracking of potentially toxic compounds used in households was possible.

Keywords: Characterisation, diary survey, greywater, inventory, screening, xenobiotic

Introduction

The awareness that centralised urban wastewater sanitation systems are considered to be expensive and resource consuming, has increased the demand in society for introduction of decentralised sanitary systems providing opportunities to save and reuse wastewater. Additionally, water shortage is a problem in several parts of the world, and one way to reduce the need for potable water is to reuse water with lower quality for non-potable purposes. There is a focus today on alternative handling of wastewater, e.g. reusing the grey wastewater. The term grey refers to wastewater from households, business complexes, hotels, schools as well as some types of industries, where no contributions from toilets, bidets or heavily polluted process water are included. This wastewater fraction has been estimated to account for approximately 75 volume-percentage of the combined residential sewage (Hansen and Kjellerup, 1994 and references therein).

One possibility for recycling grey wastewater is to use it, after treatment, for urinal and toilet flushing. Window, laundry and vehicle washing as well as fire protection and concrete production are examples of other suggested usages. Infiltration into the ground has been suggested as one alternative but grey wastewater could also be suitable for garden and crop irrigation, or to develop and preserve wetlands.

The risks related to handling and exposure to grey wastewater are major drawbacks for all the potential alternatives that are mentioned above. Spreading of diseases, caused by the exposure to micro-organisms, is of importance for almost all suggested alternatives, and due to this attention to these problems has been given in published literature and by the authorities (see e.g. Christova-Boal et al., 1996; Feachem et al., 1983). It has, however, been shown that grey wastewater can be reused for toilet flushing without a health risk if treated prior to reuse (Nolde, 1999).

Grown crops, ornamental plants, soil and receiving waters may also be affected by the content of different types of pollutants including xenobiotic organic compounds (XOCs), when grey wastewater is used for irrigation. This could e.g. lead to a deteriorating quality of groundwater and soil. However, the number of studies focusing on reuse of grey wastewater for irrigation or indirect reuse by infiltration are scarce. Health aspects and economic factors from this type of alternative wastewater handling have been examined (Nolde, 1999; Dixon et al., 1999) but no environmental risk assessment concerning infiltration has been found in the literature.

It is important to have a general characterisation of grey wastewater since the different parameters will have different impacts on human health and the terrestrial, aquatic, and agricultural environment. They will also react differently to treatment, e.g. sedimentation and biological filters. Evaluation of the appropriate treatment method should therefore be done based on knowledge of the specific characteristics. The information available regarding characteristics of grey wastewater is mainly on the content of organic matter (BOD/COD), nutrients (N, P, K) and micro-organisms, while the knowledge of the content of XOCs is limited (Eriksson et al., 2002a). The dominating source of XOCs in grey wastewater is expected to be the chemicals used in the households, e.g. laundry detergents, fabric softeners, dish-washing liquids, cleaning detergents, shampoos, soaps and toothpastes even though some small contribution of e.g. softeners from leaching pipes cannot be excluded. It is necessary to take into consideration that the characteristics of the grey wastewater will differ between

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Received 22 July 2002; accepted in revised form 29 January 2003.

different "producers". Furthermore, investigations on the importance of the composition of inhabitants (e.g. age distribution) and way of life (e.g. consumption pattern of household and personal care products) for the characteristics of grey wastewater are not available in the literature.

Nine hundred different substances or groups of substances were found to be potentially present in grey wastewater from the product information available in the list of contents of common Danish household and personal care products (Eriksson et al., 2002a). The major groups of compounds were fragrances & flavours, preservatives, solvents and some surfactants, i.e. non-ionic and anionic surfactants. Other groups were the amphoteric and cationic surfactants as well as the softeners and emulsifiers. The groups with only a few compounds in each were the bleaches, dyes, sunscreen agents and enzymes. These groups are, however, of a special interest since they all contain bioactive compounds.

The aim of this study was to collect information on the presence of the XOCs in grey wastewater. The investigations were carried out in two steps. Step one was a broad analytical screening of the characteristics of grey wastewater and step two was an inventory of the personal care products used within the households. The inventory aimed to summarise the load of chemical compounds that potentially could be present in the grey wastewater by reviewing all lists of content and measuring the consumption of household and personal care products.

Experimental

The method selected for characterisation consisted of a GC-MS screening in order to identify organic substances followed by quantitative analyses of selected parameters. Interviews and questionnaires with diary sheets were used to measure the grey wastewater-producing activities.

Case study

Sampling of grey wastewater as well as the inventory was carried out at BO90, a tenant owner's society located in the central part of Copenhagen, Denmark. The building has 17 flats, where 38 tenants are living; 22 adults (age 18 to 74) and 16 children (age 2 to 15). The grey wastewater is produced in the bathrooms and originates from the showers and hand-basins with a daily production of approximately 750 ℓ. The grey wastewater is collected on site and treated within the building with the purpose of being reused for toilet flushing (BO90, 2001). The other wastewater fractions namely, kitchen, laundry and toilet are led directly to the municipal wastewater sewerage system.

Samples

All sampling took place at the inlet to the grey wastewater treatment facility. The samples were taken in glass bottles and transported at 4ºC and in the dark to the laboratory, where the analyses were conducted immediately or the samples were stored in the dark and kept ay a temperature of 4°C until analysis. Sampling took place during 4 weeks in August 2000.

Chemical characterisation

The grey wastewater was analysed:

- qualitatively by screening analyses with the purpose to identify the XOCs present; and
- quantitatively for a selected number of parameters.

The qualitative analyses included solid-phase extraction (SPE) with four different solid phases after which the extracts were analysed by GC-MS. The internal standards, 4-fluorobenzoic acid and 4-chloro-aniline were added before extractions to all samples. The neutral organic compounds were extracted and pre-concentrated on C18 (IST Isolute) and HLB (Waters Oasis) followed by sequentially eluting with hexane, hexane:diethyl ether (1:1), diethyl ether, methanol:water (1:1), methanol:water (8:2) and methanol according to a procedure described by Paxéus and Schröder (1996). Strong cation exchange, SCX, (IST Isolute) columns were used for extraction and pre-concentration of organic bases and the compounds were sequentially eluted with acetonitrile and methanolic ammonia. The volume of the organic extracts was reduced by a stream of pure nitrogen gas or by rotary evaporation to 200 μℓ whereas the water-containing extracts were evaporated to dryness and re-dissolved in 200 μℓ acetonitrile. Finally, a chromatographic standard (2-chloro-4-methyl-quinoline) was added. The organic acids were extracted and pre-concentrated on Empore anion exchange discs (Chrompack) and in-vial-derivatised with methyl iodide (Eriksson and Ledin, 2002). Blanks consisting of MilliQ-water and reagent blanks consisting of solvents that had passed through the conditioned SPE-columns were also analysed to investigate the presence of any interferences.

The GC analysis was performed on a Hewlett-Packard 6890 Series chromatograph and an HP 5973 MS detector, while the injections were performed with a Varian 8200 CX Autosampler. The GC was equipped with a HP-5MS (30 m x 0.25 mm ID) column of 0.25 mm film thickness (Agilent Technologies, Denmark). The following GC temperature program was used: 35°C held 10 min, to 280 °C at 3°C/min, then 280 °C for 20 min. The MS was operated in electron impact mode, scanning from 40 to 550 u in 0.5 s. The ion source and the transfer line temperature were set to 150°C and 230°C, respectively. Experimental identification was based on the NIST/EPA/NIH Mass Spectral Library (Version 1.1a); the library NBS75K in the Enhanced ChemStation G1701AA, spectra published in the literature and commercial reference compounds. The compounds were considered positively identified if their spectra and retention time correlated with those of an external standard or if the spectra corresponded with two spectra from the libraries (correlation 95%). Furthermore, the response of one of the internal standard was compared with the chromatographic peaks and semi-quantitative results were obtained.

Quantitative analyses were performed either in the laboratory at E&R DTU according to Danish Standard methods and methods developed at the department (Eriksson and Ledin, 2002) or by two commercial laboratories (SGAB Analytica, Sweden and ROVESTA Miljø I/S, Denmark). The analyses included 165 different compounds and summary parameters including physical, chemical and microbiological parameters. Some of the physical/chemical and microbiological parameters measured are presented in Table 1. In comparison with values from bathroom grey wastewater found in the literature, the values from BO90 are in the same order of magnitude, e.g. the oxygen-consuming summary parameters (BOD and COD) are within the range for bathroom grey wastewater found in the literature (Eriksson et al., 2002a). The contents of alkali metals and alkali earth metalloids is higher than the literature value but it should also be noted that the potable water in Copenhagen is naturally rich in alkali metals and alkali earth metalloids (calcium 97 mg/ℓ, magnesium 20 mg/ℓ, potassium 3.7 mg/ℓ and sodium 34 mg/ℓ; Copenhagen Water, 2000).

TABLE 1
Physico-chemical and microbiological characteristics of grey wastewater

Concentration (mg/l)	Bathroom (BO90)	Bathroom (literature)*
Temperature (°C)	21.6-28.2	29
pH	7.6-8.6	6.4-8.1
COD	77-240	100-633
BOD	26-130	50-300
SS	7-207	40-120
NH_4-N	0.02-0.42	<0.1-15
NO_3-N	<0.02-0.26	0.28-6.3
Tot-N	3.6-6.4	5-17
PO_4-P	na	0.9-49
Tot-P	0.28-0.779	0.11-2
Ca	99-100	3.5-7.9
K	5.9-7.4	1.5-5.2
Mg	20.8-23.0	1.4-2.3
Na	44.7-98.5	7.4-18
Total bacterial pop. (per 100 ml)	4×10^7-1.5×10^8	107-3×10^8
Total coliforms (per 100 ml)	6×10^3-3.2×10^5	103-2.4×10^7
E. Coli (per 100 ml)	<100-2800	na

*Eriksson et al., 2002a
na: not analysed

Inventory

An empirical study of the consumption of household chemicals and personal care products was carried out at BO90 during one of the sampling weeks in August of 2000. The inventory was performed by interviews, questionnaires and products registration. The inhabitants were asked to record each activity that led to grey wastewater production in the bathrooms in diary sheets. They were also asked to present all household and personal care products they used in their bathrooms. The products were registered with name, producer, list of contents and mass. A few of the containers had lost their list of contents, in those cases the product name and producer were noted, and new identical products were examined. The following week the household chemical products were re-weighed to determine the consumption during the inventory period. In a few cases, new non-weighed products had been taken into use between the two registrations. In these cases, the mass of the newly opened container was registered and compared with new identical unopened containers. The participating tenants were anonymous (for more details see Ledin et al., 2002).

It has not been possible to assess the amount of the compounds by the use of the list of contents since that information is missing on the labels. Furthermore only compounds present in levels above 1% are required to be listed (Danish EPA, 2000) which means that compounds present in relatively low concentrations will not appear on the list of contents. Furthermore, impurities in the used raw material, help-compounds used in the production and compounds used as solvents for perfumes and aromatics are not required to be listed on the labels. Fragrances and flavours as well as aromatics are only required to be reported as "perfume" and "aroma".

Results and discussion

Chemical characterisation

Qualitative screening analyses

One hundred and ninety one (191) different XOCs were identified in the extracts from grey wastewater in the qualitative screening. Identified substances were grouped into eight substance classes based on their application and their contents were semi-quantitatively assessed (Table 2).

Half of the compounds identified were long-chain fatty acids (C_5 to C_{24}) and their esters, e.g. methyl-, hexadecyl- and octadecyl-esters. The analytical basis of this analysis (SPE and GC-MS) limits the range of detection to only cover thermally stable substances with sufficient volatility to be suitable for the GC-analysis, i.e. several surfactants in household chemicals cannot be analysed in this way, e.g. PEGs (large polymers of polyethylene glycol). The surfactant group is dominated by the long-chain fatty acids, as the acid itself, as methyl ester, as amide e.g. N-(2-hydroxyethyl)-dodecanamide or as ether, e.g. 2-(dodecyloxy)-ethanol. They were found in the range from <1 µg/l to 37 µg/l (2-(dodecyloxy)-ethanol). Both nonyl- and octylphenols were present in the grey wastewater, but none of the ethoxylates was found. The group of alkyl phenols include several compounds that have come into focus since nonylphenol is an endocrine disrupter, i.e. male and female reproductive toxicant (NTP, 2001). Nonylphenol is used as a cleaning agent, softener and stabiliser but also as an intermediate in process industry (IUCLID, 2000). It can also be formed by anaerobic degradation of the non-ionic detergent nonylphenol ethoxylates (Ahel et al., 1994) Here octyl- and nonylphenol was semi-quantitatively assessed to 0.2 and 0.4 µg/l, respectively.

The emulsifiers are used to prevent separation of fats and water in a chemical product into two layers and is typically characterised by a polar and a non-polar moiety, e.g. long-chain fatty acids and their corresponding alcohols. They were found to be present in relatively high levels, e.g. hexandecanol and octadecanol were found in 64 and 117 µg/l, respectively (Table 2).

More than 40 fragrances and flavours were identified, e.g. citronellol, coumarin, eucalyptol, hexyl cinnamic aldehyde and menthol. This group is present as perfume additives in personal care products or as flavours to, e.g. toothpaste. Caffeine is present in coffee, tea and some soft drinks. p-Cresol (4-methyl phenol) can be formed by degradation of aromatic amino acids in proteins (Paxéus and Schröder, 1996) but is also used as a perfume additive. Generally these compounds were present in low levels (<10 µg/l) except for a few. Some of these compounds have also other applications in personal care products than just as perfumes, e.g. squalene, which is an animal fat, is used as both a fat and a perfume. Menthol, however, is used only as a fragrance and flavour, but is present in higher levels (33 µg/l), which reflects its extended use in e.g. toothpastes. Several of these compounds are bioactive and pose a threat to human health as they are found to cause allergies, cancer and have a teratogenic effect (Eriksson et al., 2002b).

The group "preservatives" consists of preservatives and antioxidants. Among the antioxidants found were butylated hydroxytoluene (BHT) and butylated hydroxyanisole (BHA). These substituted phenols are widely used as antioxidants and added to plastic materials and petroleum products. BHT is also used as a food additive and as an antioxidant in food packaging and soaps as well as an anti-skinning agent in paints and inks (HSDB, 2002). It is resistant to degradation, toxic to aquatic organisms and bioaccumulating, which makes it into an environmental hazard (Eriksson et al., 2002b). Here were BHT and BHA present at 4.5 and 0.5 µg/l, respectively. Among other observed preservatives were, e.g. ethyl and methyl paraben, citric acid and phenoxy acetic acid as well as triclosan. Triclosan is used as an antiseptic in a vast array of consumer products, e.g. as odour-eaters in shoes and as antibacterial agent in toothpaste (Daughton and Ternes, 1999). The semi-quantification indicates that triclosan was present at 0.6 µg/l, which correlates well with the levels found in grey wastewater from Sweden: 0.56 to 5.9 µg/l (Palmquist and Hanæus, 2001). The parabens are one of the most widely used types of antimicrobial preservatives in cosmetics, toiletries, detergents, pharmaceuticals and foods (Acme-Hardesty, 2001). The aquatic toxicity is low but they show a weak estrogenic activity (Daughton and Ternes, 1999). Salicylic acid is a hydrolytic metabolite of acetyl salicylic acid but also used as a food preservative, whereas benzoic acid is a preservative and is frequently used as food additive and in household chemicals (Daughton and Ternes, 1999).

One pesticide was found to be present: Malathion, which is used as the active ingredient in lice shampoos, which can be purchased in Danish pharmacies.

In all samples, nine softeners and plasticisers were found, the phthalates, bis(2-ethylhexyl)-, dibutyl-, diethyl-, dimethyl and mono ethylhexyl phthalate, dominate this group. Two other plasticisers similar to di-(ethylhexyl) phthalate (DEPH) in structure were identified as well as: di-(ethylhexyl) adipate (diester of hexanedioic acid) and di-(ethylhexyl) sebacate (diester of decanedioic acid). DEHP was present at 10 µg/l, whereas the other two diesters were found in the concentrations of 1 µg/l each. All other phthalates were present in the concentrations <10 µg/l in the screening. The methyl ester of hexadecanoic acid was found present at 14.2 µg/l.

The grey wastewater samples were taken during the summer period and were found to contain at least one UV-filter/sun screen agent, 3-(4-methoxyphenyl)-2-propenoic acid, 2-ethylhexyl ester also known as parasol MCX. The concentration was estimated to be 0.5 µg/l (Table 2).

The chemicals in the solvent group are dominantly alkenes and alcohols, but a few small aromatics were also identified.

It can also be noted that several miscellaneous compounds, probably indirectly attributable to household chemicals, have been identified, e.g. medicinal residuals, flame-retardants as well as the drug nicotine. Medicinal and drug residuals can be explained by excretion from humans' bodies during showering, tooth brushing and washing (present in the mouth or on the skin or excreted by urination). The medicinal acetaminophen (paracetamol) is an analgesic and anti-inflammatory agent and is used in common painkillers. The phosphorus compounds are esters of phosphoric acid and are used as flame-retardants. Tri(2-chloroethyl) phosphate and triphenyl phosphate, were detected and they have most likely been rubbed onto the skin from clothes and washed off during showering. The presence of cholesterol and coprostanol as well as other faecal sterols indicates some faecal contamination of the grey wastewater.

A few contaminants were found in the blanks and the MilliQ samples: BHT, DEHP, octadecane, octadecene, benzoic acid and three poly-siloxanes. However, since some of these compounds were also present in the grey wastewater samples, they were set to be positively detected if the area quote between the compound and the internal standard for the grey wastewater sample was more than 10 times higher than the corresponding quote of the blank.

Quantitative analyses of selected compounds

165 parameters were analysed for in the quantitative analyses: 119 organic compounds, 34 physical and chemical parameters as well as 12 microbial parameters. A few representative parameters from the major groups of detergents were included, i.e. anionic, cationic and non-ionic detergents (Table 3). The anionic detergent LAS were found to be present in the range of <25 to 450 µg/l, cationic detergents (summary parameter) were found in the range of <100 to 2 100 µg/l while the non-ionic detergents nonylphenol and octylphenol ethoxylates all were below the limit of detection. The cationic detergent in this grey wastewater predominantly derives from hair conditioners and not from fabric softeners, which otherwise is a major source for cationic detergents in household wastewater. In this instance the laundry water is diverted to the municipal treatment plant and only the hand basin and shower wastewaters contribute to the grey wastewater.

Long-chain fatty acids were found in the range of 0.3 to 15 900 µg/l (Table 3). They have not previously been quantified in grey wastewater, but have been detected in a GC-MS screening of shower wastewater from a military facility (Burrows et al., 1991). Low levels of the volatile toluene, ethylbenzene and xylenes were found, but no other substituted benzenes were detected. Seventeen chlorophenols were included in the analyses. Most of them were below the detection limit but 2,4- and 2,5-dichlorophenol were present at 0.06 to 0.13 µg/l and 2,4,6-trichlorophenol ranged between <0.02 to 0.10 µg/l whereas the concentration of pentachlorophenol (PCP) ranged between <0.02 to 0.04 µg/l. The chlorophenols are mainly used as preservatives.

The semi-quantitative results of the screening were compared with a study of grey wastewater (Palmquist and Hanæus, 2001), of municipal wastewater (Paxéus and Schröder, 1996) and of recipient (river) water (Kolpin et al., 2002). Twenty-six out of the 191 identified, had been analysed for in these studies and the concentrations were compared with those of the screening (Table 4). The compounds derive from 5 of the previously mentioned groups i.e. 4 surfactants, 9 fragrances and flavours, 4 preservatives and antioxidants, 4 softeners and plasticisers as well as 5 miscellaneous compounds.

The concentrations of aliphatic fatty acids and their methyl esters in the screening are summary values of the individual components. The free acids are 3 times higher than in wastewater, whereas the methyl esters are the same range as in wastewater. Octyl- and nonylphenol have also been found in Swedish grey wastewater and in the US rivers.

The fragrance α-methyl benzene methanol was also detected in wastewater and 4-methyl phenol (p-cresol) was found in recipients in the USA. The average concentration of 139 streams was 0.05 µg/l whereas the maximum value found (0.54 µg/l) was slightly lower than what was found in the BO90 screening. Caffeine has also been found in both wastewater and in the recipients. The levels of the fragrances camphor and citronellol are higher in this bathroom grey wastewater than in wastewater. Farnesol, geraniol and isoeugenol were found in this study to be in the same range as in wastewater (Table 4).

The preservative benzoic acid was the same concentration range as in wastewater, but no data on benzoic acid in recipient water were available. The two antioxidants BHT and BHA were, however, detected in river water, and furthermore the level of BHT was the same as found in wastewater by Paxéus and Schröder (1996). The anti-microbial triclosan was found in the same range in the screening as in grey wastewater and recipient water.

The two plasticisers bis(2-ethylhexyl) adipate and DEHP were found in a range similar to recipient data. The values from grey

TABLE 2
Xenobiotic organic compounds found in grey wastewater with semi-quantitative concentrations (µg/ℓ)

Compounds	µg/ℓ	Compounds	µg/ℓ
Surfactants		**Fragrances and flavours continued…**	
15-Octadecanoic acid	1.6	Eugenol	1.0
1-Dodecanol	11.3	Farnesol	1.0
2-(Dodecyloxy)-ethanol	37.3	Geraniol	0.8
2-(Tetradecyloxy)-ethanol	18.7	Geranyl acetone	0.6
9-Methyltetradecanoic acid	2.7	Hexadecanoic acid	76.9
Dodecanamide, N-(2-hydroxyethyl)-	0.8	Hexyl cinnamic aldehyde	0.7
Dodecanamide, N,N-bis(2-hydroxyethyl)-	14.3	Homomyrtenol	0.9
Dodecanoic acid	15.0	Hydroxycitronellol	0.2
Hexadecanoic acid, 1,2-ethanediyl ester	8.2	Indole	3.8
Hexadecanoic acid, hexadecyl ester	4.5	Isoeugenol	0.6
Nonylphenol	0.4	Linalool	15.4
Octadecanoic acid	4.2	Linalyl propanoate	1.3
Octadecanoic acid, butyl ester	0.2	Menthol	32.6
Octadecanoic acid, 2-hydroxyethyl ester	0.9	Menthone	0.9
Octadecanoic acid, 2-methylpropyl ester	0.3	Methyl abietate	1.4
p-Octylphenol	0.2	Methyl dihydroabietate	1.1
Tetracosanoic acid, methyl ester	0.6	Methyl dihydrojasmonate	3.9
Tetradecanoic acid	12.6	Phenylethyl alcohol	0.6
Tetradecanoic acid, 12-methyl-	1.8	Squalene	133
Tetradecanoic acid, 12-methyl-, methyl ester	1.8	Terpineol	1.2
Tetradecanoic acid, dodecyl ester	1.2	Tetradecanoic acid, methyl ester	3.1
Emulsifiers		Thymol	2.5
1-Dodecanamine, N,N-dimethyl-	7.4	**Preservatives and antioxidants**	
1-Hexadecanol	63.7	2-Phenoxy ethanol	24.8
1-Octadecanol	117	Acetic acid, phenoxy-	4.0
9-Octadecenoic acid	27.4	Benzoic acid	0.5
9-Octadecenoic acid, (Z)-, methyl ester	18.0	Benzoic acid, 4-hydroxy-	1.0
Cyclododecane	8.1	Butylated hydroxyanisole	0.5
Isopropyl myristate	1.6	Butylated hydroxytoluene	4.5
Octadecanoic acid, methyl ester	4.6	Citric acid	15.0
Fragrances and flavours		Ethylparaben	0.6
α-Methyl-benzenemethanol	0.1	Malathion	1.9
1-Dodecene	4.2	Methylparaben	2.6
1-Hexadecene	0.4	Octanoic acid	3.0
3-Hexanol	0.7	Phenol, 2,6-bis(1,1-dimethylethyl)-4-	
3-Hexanone	0.3	(methoxymethyl)-	0.4
4-Methoxy-benzoic acid	12.7	Salicylic acid	0.6
4-Methyl-phenol	3.1	Triclosan	0.6
6-Methyl-5-hepten-2-one	0.1	**Softeners and plasticisers**	
Anise camphor	0.5	2-Ethyl-1-hexanol	8.5
Butanoic acid, butyl ester	0.9	Bis(2-ethylhexyl) phthalate	9.8
Caffeine	0.5	Decanedioic acid, bis(2-ethylhexyl) ester	1.0
Camphor	9.1	Dibutyl phthalate	3.1
Carvone	0.5	Diethyl phthalate	4.0
Citronellol	2.8	Dimethyl phthalate	4.9
Coumarin	1.0	Hexadecanoic acid, methyl ester	14.2
Decanoic acid	1.2	Hexanedioic acid, bis(2-ethylhexyl) ester	1.0
Dihydromyrcenol	8.9	Mono 2-ethylhexyl phthalate	1.7
Dodecanal	0.9	**UV filters**	
Dodecanoic acid, methyl ester	2.2	Parasol MCX	0.5
Eucalyptol	0.1		

Compounds	µg/ℓ
Solvents	
1,13-Tetradecadiene	1.8
1,3-Dioxolane	1.7
1,8-Nonanediol, 8-methyl-	0.6
1-Decene	0.6
1-Docosene	1.6
1-Nonadecene	0.8
1-Tetradecene	0.5
2-Hexadecanol	6.1
2-Hexanol	0.3
2-Hexanone	0.6
3-Dodecene	0.4
3-& 5-Eicosene, (E)-	7.3&5.2
3-& 5-Octadecene, (E)-	0.5&0.4
4-Dodecene	0.5
4-Heptanone	1.4
7-Tetradecene	0.2
Acetamide	8.6
Cyclohexadecane	21.1
Cyclotetradecane	4.8
Decane	4.2
Dodecane	1.2
Eicosane	4.1
Ethylbenzene	2.0
Nonane	0.2
Octadecane	1.1
Sulfuric acid, dimethyl ester	0.1
Toluene	1.4
Tridecane	2.0
Xylene, m-	3.5
Xylene, o-	0.6
Miscellaneous	
β-Sitosterol	0.7
1,1-Dodecanediol, diacetate	0.8
1,2-Ethanediamine, N-ethyl-	1.2
11-Hexadecenoic acid	0.5
11-Hexadecenoic acid, methyl ester	3.7
1-Octadecene	2.4
2-Methyl-butanoic acid, methyl ester	1.8
3-Methyl-butanoic acid, methyl ester	1.5
4-Heptanone, 3-ethyl-	0.2
4-Methyl-pentanoic acid, methyl ester	1.1
7-Hexadecenoic acid, methyl ester, (Z)-	4.2
8,11-Octadecadienoic acid, methyl ester	15.5
9,12-Octadecadienoic acid, methyl ester	7.5
9-Hexadecenoic acid	18.7
9-Hexadecenoic acid, eicosyl ester, (Z)-	5.1
9-Hexadecenoic acid, methyl ester, (Z)-	31.3
9-Hexadecenoic acid, octadecyl ester, (Z)-	4.8
9-Hexadecenoic acid, tetradecyl ester	3.2
9-Octadecenamide, (Z)-	0.6
9-Octadecenoic acid, (E-), octadecyl ester	10.6
9-Octadecenoic acid, (Z)-, 9-hexadecenyl ester, (Z)-	2.9
9-Octadecenoic acid, (Z)-, 9-octadecenyl ester, (Z)-	2.0
9-Octadecenoic acid, (Z)-, octadecyl ester	7.8
9-Octadecenoic acid, methyl ester, (E)-	2.2
Acetaminophen	1.5
Acetic acid, octadecyl ester	2.5
Benzenesulfonic acid, methyl ester	1.1
Cholest-4-en-3-one	0.9
Cholest-5-en-3-one	2.4
Cholesta-3,5-diene	12.8
Cholesterol	28.6
Cholesterol acetate	4.9
Coprostanol	0.2
Decanamide, N-(2-hydroxyethyl)-	3.2
Docosanoic acid, methyl ester	0.9
Dodecanoic acid, dodecyl ester	2.1
Dodecanoic acid, hexadecyl ester	5.3
Dodecanoic acid, tetradecyl ester	3.0
Eicosanoic acid	1.3
Eicosanoic acid, methyl ester	0.6
Glycerol β-palmitate	3.8
Heptadecanoic acid, methyl ester	1.7
Hexadecanamide	0.7
Hexadecanoic acid, 14-methyl-, methyl ester	1.1
Hexadecanoic acid, octadecyl ester	3.4
Hexadecanoic acid, tetradecyl ester	5.3
Hexadecenoic acid, methyl ester	3.9
Hexanoic acid, methyl ester	10.1
Lanosta-8,24-dien-3β-ol	0.6
Nicotine	1.2
Octadecanoic acid, 2-[(1-oxohexadecyl)oxy]ethyl ester	2.8
Octadecenoic acid, methyl ester (not 9-)	9.7
Pentadecanoic acid, methyl ester	1.8
Pentanoic acid, methyl ester	1.1
Phenol, m-tert-butyl-	0.9
Propanoic acid, 2-methyl-, 1-(1,1-dimethylethyl)-2-methyl-1,3-propanediyl ester	0.5
Propanoic acid, 2-methyl-, 2,2-dimethyl-1-(2-hydroxy-1-methylethyl)propyl ester	1.1
Propanoic acid, 2-methyl-, 3-hydroxy-2,2,4-trimethylpentyl ester	0.3
Provitamin D3	3.1
Tetradecanoic acid, 9-methyl-, methyl ester	0.5
Tetradecanoic acid, hexadecyl ester	6.5
Tri(2-chloroethyl) phosphate	0.4
Tridecanoic acid, methyl ester	1.2
Triphenyl phosphate	0.5

wastewater (Palmquist and Hanæus, 2001) were slightly higher. The plasticiser diethyl phthalate in the screening was in the same order of magnitude as quoted in literature grey wastewater but higher than the levels found in the recipient.

The painkiller acetaminophen was present at 1.5 µg/ℓ, which is in the range of what has been found in the recipient. The sterols cholesterol and coprostanol were found in bathroom grey wastewater in the range found in US river water. The two flame-retardants have also been detected in river water; however, all the results were <1 µg/ℓ (Table 4).

TABLE 3
Quantitative analyses of XOCs in grey wastewater

Compound	Concentration range(µg/ℓ)	Compound	Concentration range(µg/ℓ)
Surfactants		**Chlorophenols**	
Cationic detergents (Σ-value)	<100-2100	2,3,4,5-Tetrachlorophenol	<0.02
LAS	25-450	2,3,4,6-Tetrachlorophenol	<0.02
Nonylphenol ethoxylates	<5	2,3,4-Trichlorophenol	<0.02
Octylphenol ethoxylates	<3.0	2,3,5-Trichlorophenol	<0.02
Long-chain fatty acids		2,3,6-Trichlorophenol	<0.02
Hexanoic acid	0.3-11.8	2,3-Dichlorophenol	<0.02
Octanoic acid	8.1-283	2,4,5-Trichlorophenol	<0.02
Decanoic acid	5.5-755	2,4,6-Trichlorophenol	<0.02-0.10
Dodecanoic acid	5.9-680	2,4+2,5-Dichlorophenol	0.06-0.13
Tetracanoic acid	44.4-2808	2,6-Dichlorophenol	<0.02
Hexacanoic acid	291-7020	2-Monochlorophenol	<0.5
9-Octadecenoic acid	144-15863	3,4,5-Trichlorophenol	<0.02
Octadecanoic acid	31.0-3569	3,4-Dichlorophenol	<0.02
Eicosanoic acid	19.7-189	3,5-Dichlorophenol	<0.02
		3-Monochlorophenol	<0.5
BTEXN		4-Monochlorophenol	<0.5
1-Methylnaphthalene	<4.3	Pentachlorophenol	<0.02-0.04
1,2,3-Trimethylbenzene	<9.7		
1,2,4-Trimethylbenzene	<9.5	**Phthalates**	
1,3,5-Trimethylbenzene	<9.6	Butylbenzyl phthalate	<1
2-Ethyltoluene	<9.6	Di-(2-ethylhexyl) phthalate	11-39
2-Methylnaphthalene	<4.5	Di-cyclohexyl-phthalate	<1
4-Ethyltoluene	<9.0	Diethyl phthalate	<1-13
Benzene	<1.9	Di-isobutyl phthalate	<1-3
Camphor	9.1-11.4	Dimethyl phthalate	<1
Ethylbenzene	1.9-2.1	Di-n-butyl phthalate	<1
Fenchone	<5.8	Di-n-propyl-phthalate	<1
Isopropylbenzene	<5.5	Dipentyl phthalate	<1
Methylnitrobenzene	<3.6		
n-Propylbenzene	<5.6		
Naphthalene	<4.5		
Nitrobenzene	<15		
Toluene	1.3-1.6		
Xylene, o-	0.5-0.7		
Xylene, m,p-	3.4-3.6		

Inventory

Diary survey of water-consuming activities

Eleven of the 17 flats (30 tenants) participated in the inventory. The number of persons in each flat varied from one to five and there were more females than males taking part in the investigation. The dominating age group was the 30 to 49 year old, whereas the 50 to 69 and 70+ age groups were smaller. The majority of the households were young families with children. There were some uncertainties connected with this part of the inventory since the answers from tenants seem to give a low correlation between the hours each person spends in his/her flat and the distribution of activities. An example of this is one family that have reported 41 hand-washes divided between 96 person hours whereas another family have reported 28 hand-washes per 214 person hours. In total 1,970 person hours were reported during one week, corresponding to 9.4 h in the home per person and day (and night). Hand-washing has been the most frequent activity, which is reported to have taken place 399 times during one week (with soap 279 and without soap 120). Laundry in the hand basin only took place once during this week and no dyeing of either of hair or clothes was reported (Table 5). For further details of the inventory, see Ledin et al. (2002).

Inventory of the consumption of household chemicals

Over 90 individual products were registered and weighed in the product registration. The following product groups were recognised: shampoo, hair conditioner, soap, bath oil, cleaning detergents, glass and window detergent, soapflakes, powder laundry detergents, lime deposit remover, scouring liquid, toothpaste, tooth whitener, skin cream, hair removal cream, hair styling products, deodorants, perfumes, shower gels and lice shampoo. The dominating products (in number of bottles) were shampoo and soap.

During the inventory week, 1 100 g of household chemicals was used in the bathrooms in the 11 flats (Table 6). This corresponds to about 40 g per person and week. The most frequently used products, shampoos, soaps and oral hygiene products resulted in 66% of the total consumption. Window cleaner and powder laundry detergent were on the other hand the least used during the inventory, which correlates well with the activity scheme, since only 7 occasions of disposal of cleaning water and one hand-wash of laundry took place.

Lists of content of household chemicals

From the lists of content on the registered products, 290 different organic and inorganic compound groups were found to be present. The organic compounds (237) are listed in Table 7. Organic

TABLE 4
XOCs in comparison with other water fractions

Compound	Screening (BO90)	WWTP inflow[A]	Grey water[B]	River[C] average	River[C] max
	Concentrations in µg/l				
Surfactants					
Aliphatic fatty acids C12-18	108.7	36			
Aliphatic fatty acids methyl esters C12-18	24.1	29			
Nonylphenol	0.4		2.82-5.95	0.8	40
Octylphenol	0.2		0.08-0.16		
Fragrances and flavours					
α-Methyl-benzenemethanol	0.1	1			
4-Methyl-phenol	3.1			0.05	0.54
Caffeine	0.5	37		0.1	6.0
Camphor	9.1	2			
Citronellol	2.8	1			
Farnesol	1.0	4			
Geraniol	0.8	1			
Indole	3.8	2			
Isoeugenol	0.6	<0.5			
Preservatives and antioxidants					
Benzoic acid	0.5	1			
Butylated hydroxyanisole	0.5			0.1	0.2
Butylated hydroxytoluene	4.5	4		0.1	0.1
Triclosan	0.6		0.56-5.9	0.14	2.3
Softeners and plasticisers					
Bis(2-ethylhexyl) adipate	1.0			3	10
DEHP	9.8		8.4-160	7	20
Diethyl phthalate	4.0		4.2-38	0.2	0.42
Dimethyl phthalate	4.9		<0.1		
Miscellaneous					
Acetaminophen	1.5			0.11	10
Cholesterol	28.6			0.83	60
Coprostanol	0.2			0.088	150
Tri(2-chloroethyl) phosphate	0.4			0.1	0.54
Triphenyl phosphate	0.5			0.04	0.22

A Paxéus and Schröder, 1996
B Palmquist and Hanæus, 2001
C Kolpin et al., 2002

compounds, e.g. an acid and its corresponding sodium and potassium salt, have been presented as one compound and not three as they exist in the same form in an aqueous phase. It resulted in 53 surfactants, 26 emulsifiers, 47 fragrances and flavours, 20 preservatives and antioxidants, 3 softeners and plasticisers, 4 UV-filters, 10 solvents, 11 dyes and 61 miscellaneous compounds. By far the most extensively utilised parameters/compounds were glycerine, citric acid and perfume; each registered in more than 30 products. More than half of the 290 compounds (168) were only listed on a single household or personal care product. A few of the 168 compounds are known health hazards, e.g. acrylamide are known to be carcinogenic to animals (HSDB, 2002). However, it is beyond the scope of this paper to perform a full survey regarding health hazards.

It should be noted that among the 290 parameters were 48 that contain sodium, both as an inorganic salt but also as the counter ion

TABLE 5
Distribution of water consuming activities

Activity	Number of occasions
Disposal of cleaning water	7
Dyeing of hair or clothes	0
Face wash	45
Hair wash in hand basin	1
Hand-wash of laundry in hand-basin	1
Miscellaneous	12
Shaving in hand basin/shower	14
Shower	57
Teeth-brushing	159
Wash of hands with soap	279
Wash of hands without soap	120

TABEL 6
Consumption of household chemicals during one week

Type of product	Consumed amount (g)
Cleaners	61
Deodorants	16
Glass and window cleaners	6
Hair conditioners	91
Hair styling products	19
Lime deposit removers	21
Oral hygiene products	131
Powder laundry detergents	8
Shampoos	353
Shaving foam	13
Shower gels	62
Skin care products	74
Soaps	245

to an organic acid. Thus, there might be a potential risk of e.g. soil pollution due to the relatively high percentage of salt in household products. The background levels of alkali metals are high in the potable water in Copenhagen, but by comparing the concentrations of these elements found in the grey wastewater with data from Copenhagen Water, a notable increase can be seen in the grey wastewater for potassium and sodium (see Table 1). Sodium in the potable water is at an average of 34 mg/l whereas the grey wastewater contains 45 to 99 mg/l, which corresponds to an increase of 31 to 190%, the corresponding increase for potassium is 59 to 100%.

The compounds were divided into 10 groups; surfactants (anion, non-ion, cation and amphoteric), emulsifiers, fragrances & flavours, preservatives & antioxidants, softeners & plasticisers, UV-filters, solvents, dyes and miscellaneous but also inorganic salts (Table 7).

Discussion

The result shows that not all tenants have presented all their personal care products used during the inventory since, e.g. no cosmetics (lipstick or mascara, etc.) have been reported to be in use. At one occasion, the persons performing the grey wastewater sampling noted the use of chlorine due to high odour and high electroconductivity in the samples at the treatment plant. This was later confirmed when halogenated compounds, e.g. chloroform were found to have increased drastically at this time compared to the other sampling occasions. Another example was the observed presence of the pesticide Malathion, explained as one tenant confirmed to have used lice shampoo during the sampling period. This was not recorded in the inventory, but by personal communication after the analyses were finalised. These observations illustrate that an inventory of the use of household chemicals cannot substitute a full characterisation of the compounds actually present in grey wastewater.

Comparison between the compounds found in the inventory and the ones found in the screening revealed that only 16 compounds were present on both lists. Among them were long-chain fatty alcohols and acids: 1-hexadecanol, 1-octadecanol, dodecanoic acid, tetradecanoic acid, hexadecanoic acid, octadecanoic acid and 9-octadecenoic acid. Benzoic acid, BHT, citric acid, ethyl paraben, methyl paraben, salicylic acid and triclosan, all belonging to the preservatives and antioxidants group were also present on both lists, as well as the fragrance camphor and the emulsifier isopropyl myristate. The inventory revealed that 48 types of essential oils and herbal extracts had been used to give taste and/or smell to the household chemicals. Each essential oil consists of a number of different fragrances and flavours but the list of contents do not provide data detailed enough to assess which compounds in fact are present. Several of the surfactants and emulsifiers are complex molecules containing the long-chain fatty acids as a part of their structure, but hydrolysis might degrade them into smaller structures yielding the separated fatty acids found in the screening.

Conclusions

Almost 200 different XOCs were identified in grey wastewater from bathrooms (showers and hand-basins) by a GC-MS screening. Several fragrances like citronellol, coumarin, eugenol, farnesol, geraniol, isoeugenol and hexyl cinnamic aldehyde were identified as well as some preservatives, e.g. citric acid, salicylic acid and triclosan. Other examples of compounds are the phthalates, e.g. dibutyl and dimethyl phthalate and the sunscreen agent parasol MCX. The measurements also showed that bioactive chemicals (pharmaceuticals) were present, as well as unexpected chemicals not directly deriving from household chemicals, e.g. flame retardants and drugs. The presence of among others, detergents, softeners and preservatives was confirmed by quantitative analyses

The diary survey of water-consuming activities revealed that hand-washing was the most frequent grey wastewater-producing activity, followed by teeth-brushing and showering. In the investigation of consumption of household chemicals 92 different household chemicals and personal care products were registered. The average weekly consumption of the inhabitants was about 40 g per person. The inventory of lists of content of household chemicals and personal care products registered in total 290 chemical parameters; 30 out of 38 tenants participated in the inventory, and due to this fact, the result does not rely on all the tenants and therefore not on all chemicals used during the sampling period. The study has, however, provided detailed information on the consumption of various types of household products, although information on compound level could not be assessed due to the limited data available on the list of contents. It has been shown that it is possible

TABLE 7
Xenobiotic organic compounds present on the lists of content

Compounds	Compounds
Surfactants	Coco betaine
1-Hexadecanaminium, N,N,N-trimethyl-, chloride	Dimethicone
Acetylated lanolin alcohol	Glycerine
Alkyl polyglycoside	Glycerol laurate
Anionic surfactants	Isopropyl myristate
Cocamide MIPA	Lanolin
Cocamide propylbetaine	Lecithin
Disodium laurethsulfosuccinate	Octadecanoic acid, ester with 1,2,3-propanetriol
Dodecanoic acid	Polyethylene
Fatty alcohol ethoxylate-9	Polyoxyethylenesorbitan monolaurate
Glycol distearate	Polyvinylpyrrolidone
Glycol stearate	Sodium 12-hydroxy-(cis)-9-octadecenoate
Jojoba oil	Sorbitan mono-oleate polyoxyethylene
Laureth-23	Sorbitan monostearate
Laureth-4	Sorbitan palmitate
Laureth-7	Sorbitol
Lauryl betaine	Steareth-100
N,N-Bis(2-hydroxyethyl)cocoamide	Steareth-30
Nonionic detergents	Xanthan gum
Octadecanoic acid	**Fragrances and flavours**
Palm kernel sodium soap	1-Hexadecanol, acetate
Panthenol	*Aesculus hipocastanum* extract
PEG-12	Almond oil
PEG-150 distearate	*Aloe barbadensis* extract
PEG-20 stearate	*Ananas sativus* extract
PEG-200 hydrogenate glyceryl palmiate	Aroma
PEG-3 distearate	Bisabolol
PEG-40	*Bruxus chinensis* extract
PEG-40 hydrogenate	*Calendula officinalis* extract
PEG-40 hydrogenate castor oil	Camomile extract
PEG-60 hydrogenate castor oil	Camphor
PEG-7-glycerylcocoate	*Chamo milla recutita* extract
PEG-8	*Citrus auratium bergamia* extract
Polyquarternium-10	*Citrus auratium dulcis* extract
Polyquarternium-16	*Citrus nobilis* extract
Polyquarternium-7	*Cucuneis sativus* extract
PPG-3-methylether	Cypress bark oil
Propylenglycol	*Elaeis guineesis* extract
Quarternium-18	*Equisetum arvense* extract
Quarternium-80	Essential oils
Sodium C12-13 Parethsulfate	Herbal extract
Sodium coco soap	Isoamyl
Sodium hexadecyl sulfate	*Lamium album* extract
Sodium laureth	*Lonicea caprifolium* extract
Sodium laureth sulfate	Marcussen herbal mix no. 245
Sodium laureth-11 carboxylate	Methyoxy cinnamic acid
Sodium lauryl ether sulfate	Moroccan mint oil
Sodium laurylsulfate	*Nasturtium officinalis* extract
Sodium tallow soap	Oak bark oil
Sodiumlaureth-14 carboxylate	*Olea europaea* extract
Surfactants	Parfume
Tetradecanoic acid	*Passiflora incarnata* extract
Triethanolamine lauryl sulfate	*Persea gratissima* extract
Emulsifiers	*Prunus armeniaca* extract
1-Hexadecanol	*Pinus malus* extract
1-Octadecanol	Rasul extract
9-Octadecenoic acid	Rose jasmine extract
Acrylate	*Rosemarinus officinalis* extract
Acrylamide	*Salvia officinalis* extract
Carbomer	*Sambucus nigra* extract
Carrageenan	*Solum diatomea* extract

Compounds	Compounds
Fragrances and flavours continued… *Spiraea ulmaria* extract Stinging nettle extract Thyme *Tilia vulgaris* extract *Tritium vulgar* extract *Viola tricolor* extract *Vitis vinifera* extract **Preservatives and antioxidants** Benzoic acid, sodium salt Butylated hydroxytoluene Butylparaben Citric acid DMDM hydantoin Ethylparben Imidazolidinyl urea Isobutylparaben Methylchlorisothiazolinone Methylisothiazolinone Methylparaben Phenoxyethanol Propyl 3,4,5-trihydroxybenzoate Propylparaben Salicylic acid Sodium methylparben Sodium PCA Sorbic acid, potassium salt Triclosan Zinc PCA **Softeners and plasticisers** Castor oil, hydrogenated EDTA Triethylcitrate **UV-filters** Benzophenone 3 Benzophenone 4 p-Aminobenzoic acid UV-filters **Solvents** 2-Propanol Butane Butylglycol Ethanol Isobutane Isopentane Mineral oil Pentane Propane Triethanolamine **Dyes** Acid Blue 80 C.I. pigment green 7 Dye Fast Yellow G FD&C Blue No. 1 FD&C Yellow No. 5 FD&C Yellow No. 6 Orange II Quinoline Yellow Resorcin Brown SX Purple **Miscellaneous** 2-Hydroxypropyl methyl ether 2-Oleamido-1,3-octadecandiol	9,12,15-Octadecatrienoic acid Acrylate crosspolymer Amino methyl propanol Amyloglucosidase Calcium glycerophosphate Carboxymethyl cellulose Cellulose gum Cocoimino dipropionate Coconut oil, estrified Collagen Copolymer Corn glucose Creatine, hydrate Decyl glucoside Diethylenetriaminepentaacetic acid, pentasodium salt Dimethicone copolyol Dimethylaminoethylmethacrylate copolymer Disodium cocoamphodiacetate Disodium cocoylglutamate Disodium lauroamphodiacetete Elastin, hydrate Etridronic acid, sodium salt Formic acid, sodium salt Glucoseoxidase Glycine soya Guarhydroxypropyltrimoniumchlorid Hexadecanoic acid Hydroxyethylcellulose Hydroxypropyl guarhydroxypolytrimonium Hydroxystearylcetylether Iodopropylbutylcabamate Lactic acid Lactoperoxidase Lauryl glycoside Laurylmethylgluceth-10- hydroxypropyldimoniumchlorid Laurylpolyglucose MEA laurylsulfate Methyldibromo glutaronitrile Milk proteine Niacinamide Octyldodecanol Palm kernel acid Panthenylethylether Paraffinum Phytantriol Retinyl palmitate Rosin acid, sodium salt Saccharine, sodium salt SD alcohol 39-C Sodium cocoylglutamate Sodium laurylsarcosinate Sodium methidronate Sodiumstyrene/acrylate copolymer Stearamidopropyl dimethylamine Tallow fatty acids Tocopheryl acetate Trideceth-7 Carboxylic Acid Wheat proteine, hydrate Xylitol

to track the potentially toxic compounds used in households that may present a problem in relation to, e.g. infiltration of grey wastewater. However, the observations made in this study illustrate that an inventory of the use of household chemicals, cannot compensate for a full characterisation of the compounds actually present in grey wastewater.

Acknowledgements

The authors would like to acknowledge research assistant Morten Smith and Mrs Margrethe Sørensen for their participation in the project. Financial support from the Danish Environmental Protection Agency and the Technical University of Denmark is gratefully acknowledged.

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