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Research article

Decentralised black water treatment by combined auto-thermal aerobic digestion and ammonia – A pilot study optimising treatment capacity

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ABSTRACT

Partial heating of black water by auto-thermal aerobic digestion was combined with the addition of 1% w/w urea and monitoring of pathogens and indicator organisms over a 21-day period. After initial mixing, the 160 m^3 black water (60 m^3 heated and 100 m^3 non-heated) was left undisturbed. The urea was confirmed to be fully degraded into ammonia (5.1 g N L^{-1}) first after 14 days, while the pH stabilised at around 9.2 after one week. The initial temperature of 17 °C fell by 6 °C during the study. *E. coli* and *Salmonella* spp., which are sensitive to ammonia, were inactivated during the first few days of the study, despite the urea only being partly hydrolysed. At day 14, f-RNA bacteriophages could also no longer be detected. The more persistent somatic coliphages, *Enterococcus* spp. and *Ascaris* eggs, showed significant but slow inactivation. The treatment proved to be efficient with regards to salmonella, which is a target pathogen in the Swedish context, but for parasite egg inactivation a higher temperature was required. The treatment would benefit from more frequent stirring to speed up the hydrolysis of urea and thus improve treatment efficiency. The alternative treatment scheme could increase capacity by 2.4 times, albeit with a 40% higher cost per volume due to the increased use of urea.

1. Background

The collection and recycling of nutrients from human excreta to agricultural land would decrease environmental pollution from on-site sanitation systems and reduce the use of fossil-based chemical fertilisers (Spångberg et al., 2014; Winker et al., 2009). If low flush toilets are used, no change to in-house practice would be required, which could increase user acceptance, in contrast to source-separating, dry sanitation solutions. Thus it is likely that a future sanitation reuse scenario would involve the use of black water. However, black water potentially contains pathogens, making sanitising treatments a requirement for safe nutrient reuse.

Regulations and guidelines regarding hygiene for sewage fractions applied to agricultural land generally set either a concentration limit for the end product and/or require a pre-determined reduction in organisms to validate the treatment process (WHO, 2006). In Sweden there are currently no such regulations, but at the request of the government, the Swedish Environmental Protection Agency (SEPA) published a proposal for regulation in 2013 (SEPA, 2013). In the meantime, source-separated black water from households can be certified according to a voluntary certification, SPCR 178, which regulates traceability and product quality, including hygienic quality (SP, 2012).

Ammonia has been proven to be efficient at inactivating many groups of pathogenic microorganisms, and sanitisation has been evaluated for source-separated faeces, sewage sludge and manure (Nordin et al., 2009b; Ottoson et al., 2008; Pecson et al., 2007). Adding ammonia in the form of urea is favourable for working conditions and worker safety, compared with the use of aqueous ammonia. To sanitise black water with low dry matter, a combination of auto-thermal aerobic digestion (ATAD) up to 40 °C and subsequent ammonia treatment by adding 0.5% urea to the ATAD reactors has been developed (patent WO 2012/115589 A1; Nordin and Vinneras, 2015), and applied in the Hölö plant, located to the south of Stockholm, Sweden, since 2012. By combining ATAD with ammonia treatment, the ATAD-induced temperature elevation gives higher ammonia sanitisation rates while using less urea compared to ammonia treatment at ambient temperatures. Sanitisation with a mechanism other than heat treatment has

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eliminated the need to add complementary energy-rich material to the black water. However, the total retention time for the treatment in the combined ATAD and ammonia reactors is two weeks, causing a bottleneck in terms of the system's capacity.

The objective of this study was to evaluate the possibility of having an efficient and low-cost combined ATAD and ammonia treatment by heating some of the black water in the ATAD process. Optimisation of the treatment capacity by heating only part (60 m^3 of 160 m^3) of the black water and adding a greater amount of urea (1% w/w) in one of the collection wells instead of in the reactors was tested. The main focus was on the sanitising effect on pathogens (*Salmonella* spp. and *Ascaris suum* eggs) and indicator organisms (*Escherichia coli*, *Enterococcus* spp. and bacteriophages), with the aim of enabling safe fertiliser products to be produced from source-separated toilet waste.

2. Methods

This study was conducted as two separate batch sets (March 2016–May 2016) using source-separated black water collected from sealed septic tanks in on-site sanitation systems, some of them using vacuum toilets. Both reactors were used to aerate and heat 2*30 m^3 black water to ≥40 °C, the temperature that can be achieved by composting this substrate. The heated black water was mixed for 1 h with 100 m^3 raw black water and 1600 kg urea (1% w/w) (Yara, Sweden), and stored in one of the 200-m^3 collection basins. The temperature was monitored with Tiny tag Aquatic 2 probes (Intab, Sweden) on the surface (0 m), in the middle (1.6 m deep) and at the bottom (3.2 m deep). *Ascaris suum* eggs from sieved swine faeces (Excelsior Sentinel, Inc, USA) were added to the black water in permeable nylon bags (mesh 28 μm) (Bigman AB, Sefar) held in perforated 1-L outer containers (0.7 mm mesh) at the same depths as the temperature probes.

At the start and the end of the experiment, the black water was analysed for total solids (105 °C, 24 h) and volatile solids (550 °C, 4 h). During the study, triplicate 100-mL samples and one bag of *Ascaris* eggs were taken at each depth. After sampling, the egg-containing nylon bags were kept in 0.1 N sulphuric acid, both during cold transport and at the laboratory when incubated at 28 °C for 30 days to allow larval development (Arene, 1986). Viability counts were performed under the microscope. Eggs developing to the larval stage were considered viable, while pre-larval stages were not. Initial viability of the *Ascaris* eggs was 75% (95% confidence interval (CI_{95}) 71–77%). The pH was analysed with a glass electrode PHC 2011 (Radiometer Analytic, France) connected to a PHM 210 meter (MeterLab, Denmark) in undiluted samples that were allowed to adjust to room temperature (23 °C). For total nitrogen analysis, samples were digested with oxidising agents (Spectroquant® 114963; Merck, Germany) in a thermoreactor (TR 420 and TR 320; Merck, Germany), followed by an analysis of the nitrate formed (Spectroquant® 109713; Merck, Germany). Total ammonia nitrogen analyses (Spectroquant® 100683; Merck, Germany) were performed on filtered black water (Filtropur 45 μm, Sarstedt AG & Co, Sweden). Colorimetric readings were performed in a Spectroquant® NOVA 60 (Merck, Germany).

Salmonella spp. was analysed by pre-enriching 50 mL black water diluted tenfold in Buffered Peptone Water (BPW) (18 h at 37 ± 1 °C), followed by selective enrichment on Modified Semi-Solid Rappaport-Vassiliadis (MSRV) agar supplemented with 1.0% Novobiocin (41.5 ± 0.5 °C). Suspected *Salmonella* spp. growth, checked after 24 and 48 h, was further investigated by Xylose Lysine Deoxycholate agar (XLD) with 1.5% Novobiocin, triple sugar iron agar tubes and urease broth.

For enumeration, tenfold dilution series of samples with buffered NaCl peptone water with surfactant Tween (pH 7) (SVA, Sweden) were performed. Total coliform analysis specifying the fraction of *E. coli* was performed on Chromocult Coliform agar (Merck, Germany) using Coliform Selective Supplement when analysing low dilutions. *Enterococcus* spp. was detected on Slanetz-Bartley (SlaBa) agar (Oxoid) (48 ± 2 h at 41.5 ± 0.5 °C). F-specific RNA phages and somatic coliphages were detected by standards ISO10705-1:1995 and 10705-2:2000, using the double-layer agar method (17–24 h at 37 ± 1 °C,) with *Salmonella* Typhimurium WG49 (ATCC® 700730™) and *E. coli* 13706 (ATCC® 13706™) used as bacterial host strains for the respective bacteriophages. Samples for phage analysis were filtered (0.45 μm) to reduce the potential of disturbing bacterial growth. Phage ϕx174 (ATCC® 13706-B1™) and f-RNA phage MS2 (ATCC® 15597-B1™) were used as positive phage controls. The concentration of ammonia (NH_3) was calculated from TAN concentration, pH and temperature, according to Emerson et al. (1975).

To estimate the energy need for different treatment alternatives, data from plants using the same type of reactor (Eveborn et al., 2007; Norin et al., 2000) were used, assuming a linear temperature increase in relation to aeration. The average energy need estimated at 0.5 kWh per degree Celsius and cubic meter also encompassed heat loss from transmission and outgoing air. A scenario with incoming material holding 10 °C and aerating it to 40 °C with 0.56 kWh per degree Celsius and cubic meter was used for estimations in Table 2. Regression analysis and single factor Anova followed by *post hoc* analysis with Tukey's Honestly Significant Difference (HSD) test (at family rate 5) were performed in Minitab 16 (Minitab Inc., US), with $\alpha \leq 0.05$ unless otherwise stated.

3. Results and discussion

3.1. Characterisation of black water

The start sample of the first set had higher total solids (TS) and volatile solids (VS), as well as a darker colour and higher viscosity, probably due to unrepresentative sampling, and was considered an outlier. Omitting this outlier, the TS of the black water, 0.26–0.37% (Table 1), was lower than that reported in black water from low flush systems evaluated by Norin et al. (1996) and Palm and Malmén (2003) (TS 0.75 and 0.4% respectively). The VS constituted less of the TS than the Swedish design value of 74% (Jönsson et al., 2005) and the 71% reported by Norin et al. (1996). Considering the design values, the TS in this study would correspond to a flush volume of 17–18 L per person per day, whereas the total nitrogen concentration of 0.7–0.8 g L^{-1} corresponds to 14–15 L per person per day. Apart from the TS and VS, the composition of the black water did not differ between the two rounds other than for the concentration of microorganisms, which were consistently higher in the second round (Table 1).

The part of the black water that was composted to 40 °C, which took 10 days, had concentrations of *Enterococcus* spp. reduced by

Table 1
Characterisation of the incoming black water given as mean ± S.D with significance between sets marked with an asterisk.

Parameter	Unit	Set 1		Set 2	
TS_{start}*	% ww	0.69	±0.15	0.37	±0.040
VS_{start}*	% of TS	82	±2.7	55	±5.4
pH	–	7.8	±0.13	7.9	±0.025
TAN	g L^{-1}	0.52	±0.010	0.51	±0.012
Tot-N	g L^{-1}	0.69	±0.066	0.82	±0.040
Salmonella spp.	per 50 g wet weight	Positive		Positive	
Enterococcus spp.*	log_{10} cfu mL^{-1}	3.9	±0.012	4.5	±0.046
*E. coli**	log_{10} cfu mL^{-1}	2.6	±0.14	3.0	±0.00
Coliphages*	log_{10} pfu mL^{-1}	3.8	±0.030	4.4	±0.061
f-RNA phages	log_{10} pfu mL^{-1}	–		2.0	±0.058