

Energy and phosphorus recovery from black water

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Abstract

Source-separated black water (toilet water) containing 38% of the organic material and 68% of the phosphorus in the total household waste(water) stream including kitchen waste, is a potential source for energy and phosphorus recovery. The energy recovered, in the form of electricity and heat, is more than sufficient for anaerobic treatment, nitrogen removal and phosphorus recovery. The phosphorus balance of a UASB reactor treating concentrated black water showed a phosphorus conservation of 61% in the anaerobic effluent. Precipitation of phosphate as struvite from this stream resulted in a recovery of 0.22 kgP/p/y, representing 10% of the artificial phosphorus fertilizer production in the world. The remaining part of the phosphorus ended up in the anaerobic sludge, mainly due to precipitation (39%). Low dilution and a high pH favour the accumulation of phosphorus in the anaerobic sludge and this sludge could be used as a phosphorus-enriched organic fertilizer, provided that it is safe regarding heavy metals, pathogens and micro-pollutants.

Keywords

anaerobic treatment, black water, biogas production, phosphorus recovery, separation at source, struvite

INTRODUCTION

New sanitation concepts are based on separation at source of household wastewater streams into grey water and black water (faeces and urine) or into grey water, urine and brown water (faeces), and have a large potential to recover the important resources energy, nutrients and water. Anaerobic treatment is regarded as the core technology for energy and nutrient recovery from source separated domestic wastewater (Otterpohl et al., 1999; Kujawa-Roeleveld and Zeeman, 2006). Previous research showed that concentrated black water (faeces and urine collected in a vacuum toilet) can be efficiently treated in a UASB (Upflow anaerobic sludge blanket) reactor at a relatively short hydraulic retention time (HRT) of 9.1 days (de Graaff et al., 2010), in a UASB septic tank at a longer HRT of 29 days (Kujawa-Roeleveld et al., 2005) or in a CSTR at an HRT of 20 days (Wendland et al., 2007). A methane production of 1.8 m³ CH₄ per m³ of black water can be achieved, which can be converted to 56 MJ/p/y as electricity and 84 MJ/p/y as heat by combined heat and power (CHP) (de Graaff et al., 2010).

The nutrients are largely conserved in the effluent of the anaerobic treatment step. Van Voorthuizen et al. (2008) reported a phosphorus removal of only 5% during anaerobic treatment of black water collected with conventional flush toilets (6L per flush), i.e. 95% was conserved in the effluent. However, for more concentrated black water, collected with vacuum toilets (1L per flush), 40% of the phosphorus was removed when applying UASB-septic tank (Kujawa-Roeleveld et al., 2005).

Worldwide, 3 million tons P/year are excreted by humans, which represents 21% of the total amount of artificial phosphorus fertilizer production (14.9 million tons P/year) (Cordell et al., 2009). Because domestic wastewater contains low amounts of heavy metals compared to industrial wastewater, relatively clean phosphorus rich products can be produced from black water, that could be reused in agriculture as a fertilizer (Winker et al., 2009) or as a raw material for the phosphorus

industry (Schipper et al., 2007).

Phosphorus is produced from mined phosphate rock, mainly for the production of fertilizers (85%, 14.9 million ton P/year) (Cordell et al., 2009). However, phosphorus rock is a limiting, non-renewable resource, and it is estimated that the global phosphate rock reserves will be depleted in 50–100 years (Cordell et al., 2009). Although there are disagreements on the total global phosphate rock reserves, for many countries these are already becoming a strategic resource (Gilbert, 2009). Mining of phosphate rock also has a large negative environmental impact because it results in the production of gypsum that is contaminated with heavy metals and radioactive elements (Wilsenach and Van Loosdrecht, 2003). A large fraction of mined phosphate rock finally ends up in the oceans, causing anoxic events and a negative effect on marine life. Clearly, to prevent a global phosphorus crisis, the focus should be much more on recycling of phosphorus (Gilbert, 2009).

This paper will show the possibilities to recover phosphorus from concentrated black water. Furthermore the recovery of resources from black water is discussed with respect to energy production and energy requirements for the treatment of black water.

MATERIALS AND METHODS

Anaerobic black water treatment in a UASB reactor

Black water (BW), collected in vacuum toilets, was obtained from the DeSaR (Decentralized Sanitation and Reuse) demonstration site in Sneek (Friesland, NL). Every two weeks jerry cans were filled with black water from the buffer tank at the demonstration site (HRT of 4 h, not cooled), transported to the lab and stored at 4 °C. The black water was used as the influent for anaerobic treatment in a UASB reactor (50L) (de Graaff et al., 2010).

Phosphorus mass balance of the UASB reactor

The phosphorus mass balance of the UASB reactor was established over the first 951 days of operation (equation 1).

$$P_{\text{influent}} = P_{\text{effluent}} + P_{\text{sludge,wasted}} + P_{\text{sludge,reactor}} \quad (1)$$

Influent (black water) and effluent samples (125 samples, taken once a week) were analyzed for total phosphorus (TP). P_{influent} and P_{effluent} were defined as the accumulative phosphorus load in the black water and effluent respectively (all in gP).

The amount of phosphorus in sludge was calculated based on the TP concentration of the sludge (16 samples of UASB BW excess sludge). $P_{\text{sludge,wasted}}$ was defined as the total amount of phosphorus wasted with excess sludge (in gP). $P_{\text{sludge,reactor}}$ was defined as the amount of phosphorus that accumulated in the UASB reactor (in gP), based on a sludge bed volume of 75% of the reactor liquid volume and the TP concentration of the sludge.

Batch tests for struvite precipitation

Stirred beaker (0.6L) batch tests (magnetic stirrer, 100 – 250 rpm) were carried out to investigate phosphate removal by struvite precipitation in the UASB effluent, by adding a magnesium solution (2M MgCl₂). Six duplo tests were carried out, all during the period between day 265 – 410 of UASB reactor operation. Varying initial molar ratios of Mg:PO₄ (1.0:1, 1.3:1 and 1.8:1) and two different initial pH's were applied (original pH and pH of 9.0, adjusted with 4 M NaOH before addition of magnesium). Samples were taken before addition of magnesium and 15, 30 and 60 minutes after addition of magnesium. Samples were filtered immediately over an ashless black ribbon paper filter (Schleicher & Schuell) and prepared for analysis of the dissolved phosphate and magnesium concentrations. Temperature, pH, redox and conductivity were measured (WTW pH/cond 340 i meter) before taking the samples.

Analyses

TP was determined according to standard methods using commercially available DrLange® test kits (Hach-Lange). Samples were diluted 20 times prior to analysis to exclude possible interference of other ions on the analysis.

The free phosphate ($\text{PO}_4\text{-P}$) concentration in membrane filtered samples (0.45 μm Cronus filter PTFE) was determined by ion chromatography (Metrohm 761 Compact IC). Total soluble phosphorus ($\text{TP}_{\text{soluble}}$) in membrane filtered samples (0.45 μm Cronus filter PTFE) was determined with ICP-OES (Inductively coupled plasma optical emission spectrometry, Perkin Elmer Optima 5300 DV).

Sludge samples were destructed using the Ethos 1 Advanced Microwave digestion system of Milestone. Two grams of sample were put into a special microwave vessel, 10 mL of HNO_3 (68%) was added and milliQ water was added up to a total volume of 30 mL. The vessels were placed into the microwave. Samples were heated to 180 °C in 15 minutes and this temperature was maintained for another 15 minutes. After cooling down, the contents of the vessels were transferred to a 100 mL flask and diluted to 1% acid for analysis of P by ICP-OES.

Total Suspended Solids (TSS) and Volatile Suspended Solids (VSS) were determined according to standard methods using the ashless black ribbon paper filter (Schleicher & Schuell).

Simulation of chemical precipitation in black water

To gain more insight in chemical precipitation that might occur in the black water and in the UASB reactor, these processes were simulated in the OLI Stream Analyzer (version 2.0, OLI Systems). A theoretical composition of black water was calculated using literature data on the composition of faeces and urine (Kujawa-Roeleveld and Zeeman, 2006; Maurer et al., 2006) and using the composition of drinking water in Friesland (www.vitens.nl). A pH survey by adding NaOH and HCl was carried out from pH 5 to 10 to determine the effect of pH on the composition of the black water. Because struvite is not present in the main database of OLI Stream analyzer, a separate database was used, provided by OLI Systems Inc. Details about chemical models, databases and about OLI Stream analyzer can be found on the website of OLI Systems.

RESULTS AND DISCUSSION

P balance UASB

Figure 1 shows the phosphorus mass balance over the UASB reactor. Only 61% of the TP left the UASB reactor with the effluent, 18% was wasted with the sludge and 8% accumulated in the reactor. About 13% of the TP was missing, which could be due to errors introduced by sampling and analysis. Furthermore some precipitates were observed on the walls and bottom of the UASB reactor, which were not wasted with excess sludge and could therefore partly explain the missing TP. The mean phosphorus content in the UASB sludge was 51 mgP/gTSS ($\text{sd}^1 = 12$), which is much higher than the typical content of bacteria cells of 2.0% (20 mgP/g dry weight, (Tchobanoglous et al., 2003)).

Concentrations in BW and effluent

Table 1 gives the measured concentrations of influent black water. The results of table 1 show that 60% of the phosphorus in black water was present as precipitates or was associated with suspended solids. Because the faeces exist for a large part of bacteria (Vinnerås, 2002), it was estimated that 67 mgP/L in first period and 54 mgP/L in the second period was organically bound in suspended solids

¹ sd = standard deviation

(using a typical phosphorus content of bacterial cells of 2.0% (Tchobanoglous et al., 2003)). This corresponds with 52 – 59% of the TP associated with solids. The remaining phosphorus in the suspended solids could be mineral calcium phosphates originating from faeces (Vinnerås, 2002) and other precipitates from urine such as struvite (Udert et al., 2003), but this was not further quantified.

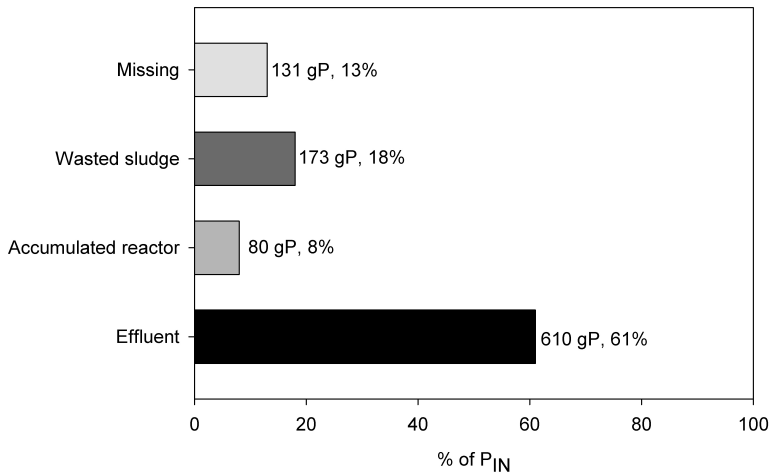


Figure 1. Phosphorus balance of the UASB reactor treating concentrated black water over the first 951 days of operation; numbers are the accumulative load of phosphorus (The total amount of TP fed to the reactor during this period was 994 gP).

Most of the soluble phosphorus (TP_{soluble}) was present as free phosphate (PO₄-P), which was 36% of TP_{IN} (table 1). When assuming that all phosphate from urine was excreted in soluble inorganic form (Vinnerås, 2002), a much higher PO₄-P concentration of 107 mg/L was estimated. During transport and storage of black water free phosphate from urine may have precipitated, resulting in relatively low PO₄-P concentrations in the black water, respectively 79 mg/L and 54 mg/L in the first and second period. Research on black water with a dilution factor of 21, showed a PO₄-P concentration of 27 mg/L (van Voorthuizen et al., 2008), which corresponds well with the estimated value in of 25 mgP/L, assuming that all phosphate from urine was excreted in soluble inorganic form (Vinnerås, 2002).

Table 1 Measured characteristics of influent concentrated black water

	unit	UASB Influent (concentrated black water) (125 samples)				% of TP _{IN}
		1 st period (day 1 – 518)	sd	2 nd period (day 519 – 951)	sd	
pH	-	8.8	0.22	8.6	0.53	
TP	mg/L	220	67	148	64	
TP _{SS} = TP – TP _{soluble}	mg/L	130	-	91	-	60%
TP _{soluble}	mg/L	90	8.7	57	18	40%
PO ₄ -P	mg/L	79	8.5	54	27	36%
VSS	g/L	3.3	1.4	2.7	1.4	

sd = standard deviation

During anaerobic treatment the free phosphate concentration increased from 79 mgPO₄-P/L to 92 mgPO₄-P/L in the first period and from 54 to 69 mgPO₄-P/L in the second period (tables 1 and 2). This probably was caused by hydrolysis of suspended solids containing organically bound phosphorus and by a decrease in pH during anaerobic treatment (from 8.6 – 8.8 in black water to

7.4 – 7.6 in UASB effluent, tables 1 and 2) solubilising inorganic phosphates. The effluent contained 61% of the TP in the influent black water, of which the main part was present as free phosphate (44% of the influent load of TP). A small fraction of the phosphorus left the UASB reactor associated with solids in the effluent, on average 14% (table 2).

Table 2 Measured characteristics of the UASB effluent

	unit	UASB Effluent (125 samples)				% of TP _{IN}
		1 st period (day 1 – 518)	sd	2 nd period (day 519 – 951)	sd	
pH	-	7.6	0.13	7.4	0.17	
TP	mg/L	131	15	94	18	61%
TP _{SS} = TP – TP _{soluble}	mg/L	28	-	24	-	14%
TP _{soluble}	mg/L	103	10	70	11	47%
PO ₄ -P	mg/L	92	11	69	13	44%
VSS	g/L	0.15	0.12	0.053	0.066	

sd = standard deviation

Simulation of chemical precipitation in black water

Low dilution and high pH seem to play an important role regarding the availability of free phosphate in the black water and during anaerobic treatment for subsequent phosphorus recovery by struvite precipitation. Both aspects were further investigated by simulating this chemical precipitation in the black water.

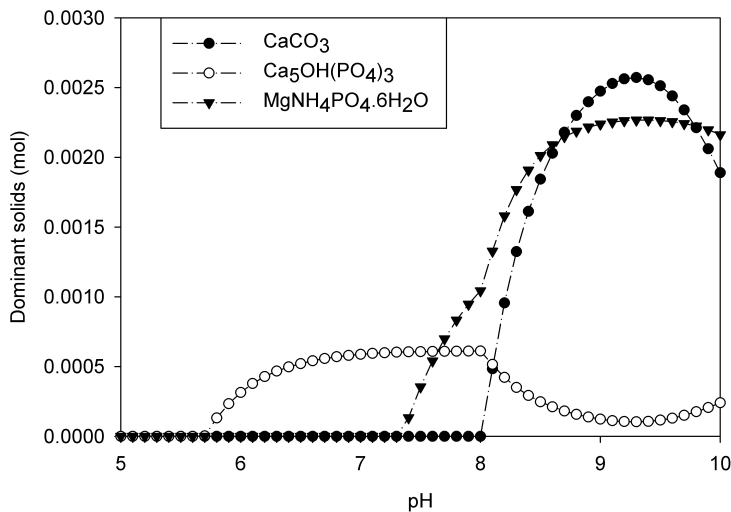


Figure 2 The predicted effect of pH in concentrated black water on the precipitation of calcite (CaCO₃), hydroxyapatite (Ca₅OH(PO₄)₃) and struvite (MgNH₄PO₄·6H₂O).

The results in figure 2 clearly show that struvite is likely to precipitate when the pH in black water is higher than 7.4. Because the pH of the black water used in this research attained values of 8.6 – 8.8 (table 1), struvite and hydroxyapatite will likely precipitate during storage and transport. It should be mentioned that kinetics are not taken into account in the simulation, which will have an effect on the type of precipitates formed (Musvoto et al., 2000). Therefore, the results from the simulation are indicative, showing the possible effect of the amount of flushing water and pH. This was also shown for the separate collection of urine, where the risk of blockages can be diminished by urine dilution (Udert et al., 2003). Similar effect of pH can be expected for the phosphorus

distribution in the UASB reactor.

The composition of tap water will also have a large effect on the concentrations in black water, because the tap water that was used in this research was relatively soft compared to tap waters in other regions or countries (e.g. tap water in Germany contains up to 199 mg/L of calcium). The use of hard tap water will result in more precipitation of calcium phosphates in concentrated black water, reducing the availability of PO₄-P in the anaerobic effluent.

Struvite precipitation

Batch precipitation tests were carried out at different pH values (8 or 9) and at different initial molar ratios of magnesium to phosphate. The results in table 3 show that the removal efficiencies of phosphate depended on the applied pH as well as the surplus of magnesium that was added. Either by adding a surplus of magnesium (e.g. Mg:PO₄ 1.5:1 at pH 8.0) or by adjusting the pH to 9.0 at a lower Mg:PO₄ ratio of 1.3:1, a removal efficiency of 90% could be achieved, which was similar as in other research reviewed by Le Corre (2009) (60-98%). A higher Mg:PO₄ ratio of 1.8:1 did not significantly contribute to an improved removal efficiency (table 3). Using these results, 0.15 kg PO₄-P/p/y can be recovered in the form of struvite (based on a PO₄-P concentration of 92 mg/L and a black water production of 5 L/p/d). Because together with the struvite, suspended solids will be removed as well, a maximum of 0.22 kgP/p/y can be recovered from UASB effluent (based on 131 mg TP/L). Obviously, latter will reduce the purity of the produced struvite. The recovered struvite would represent 10% of the artificial phosphorus fertilizer production worldwide (14.9 million tP/year, (Cordell et al., 2009)).

Table 3 Phosphate removal efficiencies in UASB effluent by the addition of different amounts of magnesium and at different pH after 60 minutes

molar ratio Mg : PO ₄	pH 8.0 (sd)	pH 9.0 (sd)
1.0:1	72% (3.5%)	82% (8.2%)
1.3:1	-	91% (0.7%)
1.5:1	90% (0.0%)	96% (0.7%)
1.8:1	-	92% (0.7%)

Precipitation of struvite from urine showed that the struvite can be recovered free from organic micro-pollutants and free from most of the heavy metals present in urine (Ronteltap et al., 2007). More research with respect to micro-pollutants in struvite recovered from UASB effluent is needed, because the co-precipitated solids and organic material may contain adsorbed micro-pollutants.

The main challenges to make struvite precipitation for phosphate recovery a successful technology, are the quality of the struvite and the economic value of the struvite (Le Corre et al., 2009). In Japan struvite produced from digester liquor was successfully sold to fertiliser companies (Ueno and Fujii, 2001). Recently the struvite produced from an industrial wastewater treatment plant treating potato wastewater in Steenderen (NL) is being used by a company producing cattle fodder², showing an increasing interest for struvite as a valuable product.

Energy recovery from black water

The amount of electricity that can be produced is significant in terms of wastewater treatment. Whereas conventional wastewater treatment plants consume 96 MJ/p/y or 26.6 kWh/p/y (Frijns et al., 2008), from the produced methane in the black water treatment concept 56 MJ/p/y can be recovered as electricity and 84 MJ/p/y as heat. An estimation of the energy production and consumption in the black water treatment concept is presented in table 4, and is compared with

² http://www.netserver2.net/waterforum/index.asp?url=/template_a1.asp&que=paginanr=4894, news item in Dutch, 31st March 2009

conventional wastewater treatment. In the estimation nitrogen removal from black water is included, which can be achieved with the energy efficient process of partial nitrification-anammox (Vlaeminck et al., 2009; de Graaff et al., submitted).

Table 4 shows that 22.5 MJ/p/y of electricity is gained in the black water treatment concept, whereas in sewerage and conventional WWTPs 107 MJ/p/y is lost. These numbers provide an indication, because energy needed for the production of hardware such as reactors and pumps etc. and heat losses are excluded. Table 4 shows that the electricity that is produced from black water can more than compensate for the electricity demand of nitrogen removal and phosphorus recovery. Furthermore, by using less drinking water for toilet flushing, 25% of the total drinking water consumption in a household can be saved, which results in a further saving of about 19 MJ/p/y (table 4). More savings could be achieved by reusing the struvite as a P fertilizer, requiring less fossil fuels for synthetic fertilizer production. This was however not further quantified.

Table 4 Energy production and consumption in the black water treatment concept and conventional WWTPs.

Black water (5 L/p/d)	MJ/p/y electricity	MJ/p/y heat	Conventional WWTP, domestic sewage (124 L/p/d)	MJ/p/y
Vacuum transport ^a	-14		Sewerage ^f	-43
BW heating ^b		-80	WWTP ^f	-96
Biogas production ^c	+56	+84	Biogas production from sludge digestion ^f	+32
P removal by struvite precipitation ^d	-3.5			
N removal by nitrification- anammox ^d	-16			
Total	+22.5			-107
Savings: Drinking water production ^e	+19			

^a4 kWh/p/y, most energy efficient vacuum toilet (WRS, 2001); ^bincrease of 9 degrees (worst case), transfer efficiency of 85%; ^c12.5 L CH₄/p/d, 35.6 MJ/m³ CH₄, 85% efficiency (40% electricity and 60% heat); ^d(Maurer et al., 2003); ^e0.47 kWh/m³ (Vewin, 2006); ^fIncluding N and P removal, (Frijns et al., 2008)

CONCLUSIONS

New sanitation concepts, based on separation at source of household wastewater streams have a large potential to recover the important resources energy, nutrients and water. Anaerobic treatment of black water, as the core technology for energy and nutrient recovery, provides sufficient energy for the combined anaerobic treatment, nitrogen removal and phosphorus recovery. In the anaerobic effluent of a UASB reactor treating concentrated black water, 61% of the phosphorus was conserved. Phosphate can easily be removed from anaerobic effluent by struvite precipitation, recovering 0.22 kgP/p/y, which represents 10% of the artificial phosphorus fertilizer production in the world. The remaining phosphorus ended up in the anaerobic sludge due to the low dilution factor. This sludge might be used as a phosphorus-enriched organic fertilizer, provided that it is safe regarding heavy metals, pathogens and micro-pollutants.

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