

Article

Improved Energy Recovery by Anaerobic Grey Water Sludge Treatment with Black Water

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Abstract: This study presents the potential of combining anaerobic grey water sludge treatment with black water in an up-flow anaerobic sludge blanket (UASB) reactor to improve energy recovery within source-separated sanitation concepts. Black water and the mixture of black water and grey water sludge were compared in terms of biochemical methane potential (BMP), UASB reactor performance, chemical oxygen demand (COD) mass balance and methanization. Grey water sludge treatment with black water increased the energy recovery by 23% in the UASB reactor compared to black water treatment. The increase in the energy recovery can cover the increased heat demand of the UASB reactor and the electricity demand of the grey water bioflocculation system with a surplus of 0.7 kWh/cap/y electricity and 14 MJ/cap/y heat. However, grey water sludge introduced more heavy metals in the excess sludge of the UASB reactor and might therefore hinder its soil application.

Keywords: energy recovery; anaerobic treatment; grey water sludge; black water; heavy metals

1. Introduction

Biogas as a renewable energy source is increasing its energy market share with the enforced regulation of organic waste treatment and recycling in the European Union (EU) [1]. Co-digestion is a well-known concept for improving the biogas yield from different wastewater streams and biosolids due to positive synergisms in the microbiota, the supply of nutrients, the dilution of inhibitive compounds and the improved moisture content of the feed [2]. Several studies have investigated the co-digestion of different substrates, such as sewage sludge and grease trap sludge [3,4], cattle slurry and cheese whey [5], potato processing water and glycerol [6] and a number of different animal manures and energy crops, also mentioned in the white paper of renewable energy sources (RES) of the EU-Commission from 1997 [7].

Anaerobic treatment of source-separated domestic wastewater is recognized as the core technology to improve energy recovery from domestic wastewater [8]. Concentrated black water (toilet water) and kitchen refuse has been traditionally considered as the main source for energy recovery in the decentralized sanitation and reuse (DESAR) concept [9]. Co-digestion of kitchen refuse with black water has shown improved methane production in both accumulation systems [10] and UASB septic tanks [11]. Grey water originating from washing activities in the household, however, contributes a significant fraction of the organic load present in domestic wastewater. Currently, this fraction is lost either by using a constructed wetland or a sequencing batch reactor (SBR) [12], where the organic matter is oxidized instead of utilizing it as an energy source. Alternatively, the organic fraction can be concentrated in a bioflocculation unit, such as the membrane bioreactor (MBR) or the A-trap of the Adsorption-Belebung (AB)-process [13] and subsequently treated with black water in an up-flow anaerobic sludge blanket (UASB) reactor for improved energy recovery. In the study of Hernández Leal *et al.* [14], grey water was bioflocculated in an MBR, and anaerobic batch experiments on the produced sludge indicated a high biochemical methane potential (BMP) (88%).

No studies, however, have experimentally investigated grey water sludge treatment with black water in both batch and continuous UASB reactor experiments. This study presents the potential of grey water sludge treatment with black water in batch experiments by determining the BMP of black water, grey water sludge from an A-trap and their mixture. Continuous experiments are further conducted to compare the reactor performance, COD mass balance and methanization of two UASB reactors: one operated on black water and one on the mixture. In addition, the influence of grey water sludge addition on the quality of UASB reactor excess sludge in terms of heavy metals is examined to evaluate its application in soil improvement.

2. Materials and Methods

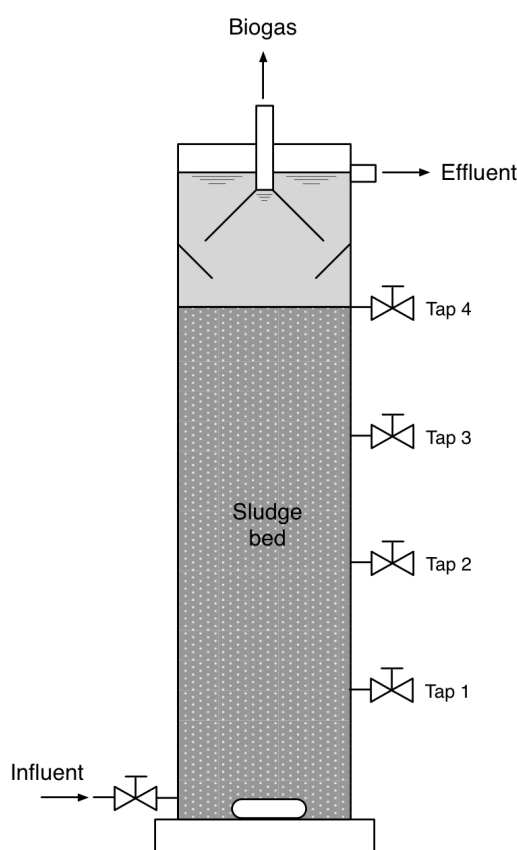
2.1. Grey Water Sludge and Black Water Source

Every two weeks, grey water sludge and concentrated black water (vacuum collected) were collected from the 32 houses in the DESAR demonstration site in Sneek, the Netherlands [14]. Grey water sludge was collected from a storage tank connected to the bottom of the settling tank of the A-trap, and black water was collected from a buffer tank. Grey water sludge and black water were transported to the experimental hall in Leeuwarden and stored in a cold room at 4 °C before feeding to the reactors.

2.2. Experimental Setup of the UASB Reactors

In this study, two 50 L UASB reactors were operated at 25 °C for 490 d on vacuum-collected black water and 498 d on the mixture of black water and grey water sludge (Figure 1). The chemical oxygen demand (COD)-based mixture ratio of 5:1 (black water:grey water sludge) was according to the actual COD loadings of these two streams at the DESAR demonstration site. Black water and grey water sludge were mixed volumetrically at the same mixture ratio of 5:1. The steady state was assumed after 90 d of operation with stable methane production. The reactors were inoculated with 20 L of anaerobic sludge (9.7 gVSS/L) from an UASB reactor operated on vacuum-collected black water at 25 °C. The details of the reactor are described in the study of de Graaff *et al.* [15].

Figure 1. The up-flow anaerobic sludge blanket (UASB) reactor configuration.



2.3. Analyses and Measurements

Influent and effluent samples (0.5–1 L) (36 samples) were collected weekly and analyzed right after collection for total suspended solids (TSS), volatile suspended solids (VSS), COD_{total} , $COD_{suspended}$, $COD_{colloidal}$, $COD_{soluble}$, total nitrogen (TN), total phosphorus (TP), anionic surfactants (AS), total ammonia nitrogen (NH_4-N), volatile fatty acid (VFA) (acetic acid, propionic acid and butyric acid), anions (PO_4^{3-} , Cl^- , SO_4^{2-} , NO_2^- and NO_3^-), soluble elemental phosphorus ($TP_{soluble}$) and inorganic carbon (IC). The details of the sample collection and analysis are described in the study of de Graaff *et al.* [15]. The AS concentration was determined with cuvette tests based on the methylene blue active substances (MBAS) standard method [16]. Biochemical oxygen demand (BOD_5) was determined from the UASB reactor effluent (4 samples). Biogas composition was analyzed from 9 samples with gas

chromatography (Shimadzu GC-2010 gas chromatograph containing GS-Q (CO₂) and HP molsieve (O₂, N₂, H₂S and CH₄) columns. Excess sludge (14 samples) and sludge bed samples taken from Taps 1, 2, 3 and 4 (3 times 4 samples) were analyzed for COD_{total}, TSS and VSS. The hydraulic retention time (HRT), sludge retention time (SRT) and the COD mass balance were calculated as described in the study of de Graaff *et al.* [15].

Total lipid, protein and carbohydrate content was analyzed from the UASB reactor influent (4 samples) and grey water sludge (1 sample). Total lipid was determined using the Bligh–Dyer extraction method from acidified samples [17] and measuring the lipids gravimetrically after the solvent was evaporated at 80 °C [16]. Total protein was determined from the difference between the corresponding TN and ammonia nitrogen concentrations and dividing the difference by 0.16 [18]. One gram of protein (assumed as (C₄H_{6.1}O_{1.2}N)_x) was considered equal to 1.5 g COD [19], and the remaining COD was termed as carbohydrates.

2.4. Batch Experiments

Biochemical methane potential (BMP), calculated as a percentage of the influent COD converted to methane, was determined from black water, grey water sludge and the mixture. The experiment was done in triplicate in 500-mL glass bottles with Oxitop heads according to the study of Kujawa [9]. The inoculum sludge used in the experiment was anaerobic sludge from a municipal wastewater treatment plant in Leeuwarden (20 gVSS/L). The bottles were placed on a shaker and incubated at 35 °C for 60 d.

2.5. Statistical Analysis

Statistical analysis of the data was done using the hypothesis testing of Statdisk. The normality of the data sets was defined according to Hair *et al.* [20] and was confirmed as normally distributed at a confidence interval of 95%.

2.6. Energy Recovery Calculations

The methane production (L/cap/d) in the UASB reactors was calculated by determining the COD load of black water and grey water sludge (86 gCOD/cap/d for black water and 15 gCOD/cap/d for grey water sludge according to the production at the DESAR demonstration site [21]) and using the fraction of incoming COD converted to methane from the COD mass balance of the UASB reactors. The volume of the produced methane was calculated using a theoretical methane production of 0.35 L/gCOD at standard temperature and pressure (STP), and the primary energy production from methane was calculated using the volume of methane and the calorific value of methane (35.8 MJ/m³) [22]. The increased heat demand of the MIX-UASB reactor was due to the heating of the additional influent stream of grey water sludge and the increased heat loss through the reactor walls of the larger reactor. The heat demand was calculated according to Tervahauta *et al.* [23] by using grey water sludge production of 1.0 L/cap/d and a temperature of 19 °C (annual average) [21], an operational temperature of 25 °C for the UASB reactor and an ambient temperature at the DESAR demonstration site of 19 °C (annual average) [21].

2.7. Heavy Metal Analysis

To assess the influence of grey water sludge addition on the quality of UASB reactor excess sludge, heavy metals were analyzed in 3 samples of grey water sludge and the influent and excess sludge of the UASB reactors. Prior to the analysis, samples were dried at 105 °C overnight and acid digested using the Ethos 1 Advanced Microwave digestion system of Milestone. Dried sample (0.5 g) was put into a special microwave vessel with 10 mL of nitric acid (68%). The samples were heated in the microwave at 180 °C for 25 min. After cooling down, the samples were diluted to reach an acid concentration of 1%. The acid destruction was done in duplicate, and the relative standard deviation (%RSD) was controlled within 20%. The samples were analyzed with inductively-coupled plasma-atomic emission spectroscopy (ICP-AES) for arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), nickel (Ni), lead (Pb) and zinc (Zn). The limit of detection (LOD) for ICP-AES was 25 ppb for Cu, Cd, Cr, Zn and Ni and 250 ppb for As and Pb. The heavy metals below these limits were analyzed with inductively-coupled plasma mass spectrometry (ICP-MS) in an external lab with an LOD of 0.02 ppb for Hg, 0.1 ppb for Cd, 1 ppb for As, Cr, Pb and Ni, 5 ppb for Cu and 10 ppb for Zn. The ICP-AES/MS analysis was done in duplicate, and the %RSD was controlled within 5%.

3. Results and Discussion

3.1. Energy Recovery

To determine energy recovery in grey water sludge treatment with black water, two UASB reactors, one operated on black water and one on the mixture (5 BW:1 GW-S), are examined for the COD mass balance and methanization. Figure 2 presents the COD mass balance of the UASB reactors over the total period of operation of 490 d for the BW-UASB reactor and 498 d for the MIX-UASB reactor. The total amount of COD fed to the reactor during this time is 21 kg, of which 3% is inoculum sludge for the BW-UASB reactor and 30 kg of which 1% is inoculum sludge for the MIX-UASB reactor. “Methane” is the amount of CH₄-COD produced in the reactor, “effluent” is the amount of total COD discharged with effluent, “sludge wasted” is the amount of COD wasted as excess sludge, including sludge bed samples, and “sludge reactor” is the amount of COD accumulated in the sludge bed. The difference between the total incoming COD and total outgoing COD is 8% for the BW-UASB reactor and 4% for the MIX-UASB reactor, and this fraction can be explained by errors in sampling and analyses. The amount of CH₄-COD produced in the MIX-UASB reactor (63%) is higher compared to the BW-UASB reactor (60%). Similarly, the BMP of the mixture (88%), determined in the batch experiments, is higher compared to black water (61%) (Table A1). The BMP of the mixture is higher than the calculated one based on the mixture ratio of 5:1 (BW:GW-S) (65%) and could be due to synergistic effects of micro-organisms increasing the biodegradable fraction in black water as a result of grey water sludge addition [24,25]. The mechanisms behind these processes, however, are not yet known.

Surfactants and lipids present in the sample can also influence the digestion process. Surfactants are known to inhibit methanogens [26], and the high AS content of grey water sludge and the MIX-UASB reactor influent (523 and 328 mg/L, respectively) compared to the BW-UASB reactor influent (189 mg/L) (Table 1) could therefore result in a decreased methane production. However, as lipids have a higher

anaerobic biodegradability compared to carbohydrates and proteins [27], the high lipid content of grey water sludge and the MIX-UASB reactor influent (74 and 59 wt%, respectively) compared to the BW-UASB reactor influent (20 wt%) (Table 1) can increase the methane production in the MIX-UASB reactor. In fact, a higher methane content of the biogas is recorded in the MIX-UASB reactor (70%) compared to the BW-UASB reactor (67%).

Figure 2. Chemical oxygen demand (COD) mass balance of the UASB reactors over the total period of operation (490 d for the black water (BW)-UASB reactor and 498 d for the MIX-UASB reactor).

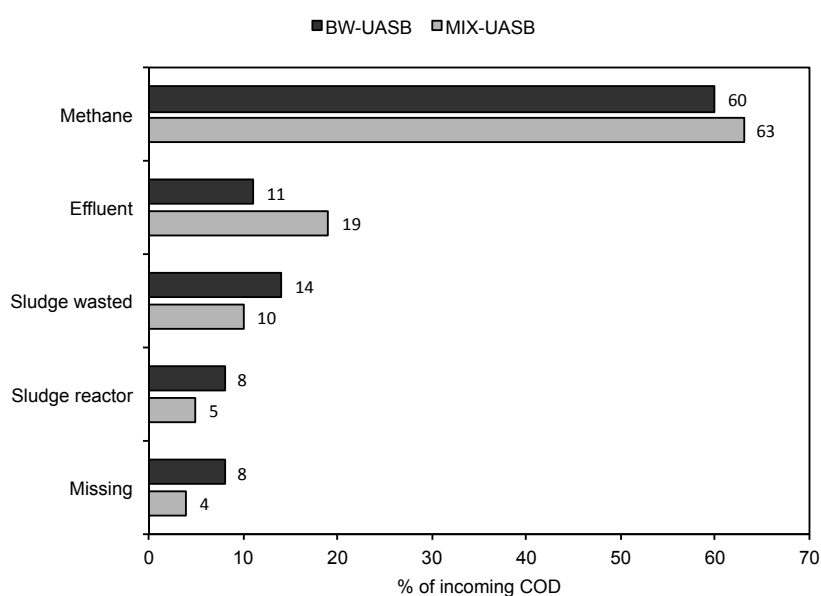


Table 1. Surfactants and organic components in the influent of the UASB reactors and the grey water sludge added to the MIX-UASB reactor. GW, grey water; AS, anionic surfactants; TSS, total suspended solids; VSS, volatile suspended solids.

Parameter	Unit	BW-UASB	MIX-UASB (5 BW:1 GW-S)	
		Influent	Influent	GW Sludge
AS	mg/L	189 (52)	328 (259)	523 (383)
TSS	g/L	4.1	6.0	8.1
VSS	g/L	3.3	4.4	6.7
COD _{total}	gCOD/L	7.1 (1.1)	17 (3.9)	63
Lipid	gCOD/L	1.4 (0.26)	10 (1.9)	47
	wt%	20	59	74
Protein	gCOD/L	4.2 (0.26)	5.6 (2.0)	10
	wt%	58	33	15
Carbohydrate	gCOD/L	1.5 (0.76)	0.8 (1.0)	6.7
	wt%	22	4.7	11

Note: () standard deviation.

The amount of COD discharged with effluent is higher in the MIX-UASB reactor (19%) compared to the BW-UASB reactor (11%) and might be due to an occasional washout of sludge to the effluent. The amount of COD in the wasted sludge and accumulated in the sludge bed are lower in the MIX-UASB reactor (10% and 5%, respectively) compared to the BW-UASB reactor (14% and 8%, respectively) and can be attributed to the higher methane production and the occasional loss of sludge with the effluent in the MIX-UASB reactor.

The two UASB reactors are operated with the aim of applying the same conditions for comparing their performance. Table 2 presents the operational conditions and methanization of the UASB reactors at steady state defined as the period of stable methane production after 90 d of start up. The HRT is slightly higher in the MIX-UASB reactor, but does not represent a significant difference at a confidence interval of 95%. The loading rate, however, is higher in the MIX-UASB reactor (1.2 compared to 0.9 kgCOD/m³/d), due to fluctuations in the influent COD concentration resulting from the grey water sludge addition. Nevertheless, the operational conditions in both UASB reactors are considered comparable. The sludge bed in both UASB reactors is compact and well developed, resulting in a similar sludge concentration and SRT. The methanization in the UASB reactor is calculated by dividing the total amount of methane-COD produced with the COD_{total} removed in the reactor. The methanization in the UASB reactor is 5% higher with grey water sludge treatment. Calculated based on the applied HRT, black water production of 5 L/cap/d and grey water sludge production of 1.0 L/cap/d [21], the volume of the reactor at full scale would be 46 L/cap for the BW-UASB reactor and 60 L/cap for the MIX-UASB reactor, resulting in 14 L/cap higher reactor volume for grey water treatment with black water.

Table 2. Operational conditions and methanization of the UASB reactors at steady state. HRT, hydraulic retention time; SRT, sludge retention time.

Parameter	Unit	BW-UASB	MIX-UASB
Temperature	°C	25	25
Loading rate	kgCOD/m ³ /d	0.9 (0.2)	1.2 (0.5)
HRT	d	9.3 (0.9)	10 (2.2)
SRT	d	138 (73)	131 (90)
Sludge concentration	gVSS/L _{reactor}	15 (2.4)	15 (2.9)
Methanization	%	69	74

Note: () standard deviation.

Grey water sludge treatment with black water increases the energy recovery in source-separated sanitation concepts by introducing an additional organic fraction (increasing the loading rate) and by increasing the methanization in the UASB reactor. The methane production in the MIX-UASB reactor is 22.2 L/cap/d (0.22 m³/kgCOD_{in}, 0.27 m³/kgCOD_{removed}, 0.26 m³/m³_{reactor}/d) and 18.1 L/cap/d (0.21 m³/kgCOD_{in}, 0.23 m³/kgCOD_{removed}, 0.18 m³/m³_{reactor}/d) in the BW-UASB reactor, representing an increase of 23%. This increase is lower compared to the theoretical one calculated in the study of [14] (73%) and could be due to the MBR used to bioflocculate grey water, producing a higher loading of grey water sludge of 40 compared to 29 gCOD/cap/d in this study. In the study of Tervahauta *et al.* [23], however, the calculated increase in the energy recovery of 28% is similar to this study using an A-trap

to biofloculate grey water. The increased energy recovery in the MIX-UASB reactor is equivalent to 55 MJ/cap/y primary energy and can cover the increased heat demand of the MIX-UASB reactor of 27 MJ/cap/y with a surplus of 28 MJ/cap/y. Considering an efficiency of 85% of combined heat and power generators, of which 40% is electricity and 60% heat, this primary energy can produce 2.7 kWh/cap/y that covers the electricity demand of the A-trap (2 kWh/cap/y) [23]. Grey water sludge treatment with black water produces an amount of energy that can cover the increased heat demand of the UASB reactor and the electricity demand of the A-trap with a surplus of 0.7 kWh/cap/y electricity and 14 MJ/cap/y heat.

3.2. Effluent Quality

To investigate the influence of grey water sludge treatment with black water on the effluent quality of the UASB reactor, the reactor performance and effluent quality of the two reactors are compared. Table 3 presents the removal efficiencies and characteristics of the influent and effluent of the UASB reactors at steady state and the characteristics of the grey water sludge added to the MIX-UASB reactor. The effluent concentrations of COD_{total} , $COD_{suspended}$, $COD_{colloidal}$, $COD_{soluble}$ and TN are similar in both UASB reactors at a confidence interval of 95%. Furthermore, the effluent concentrations of VFA, TP and BOD_5 can be considered similar in both UASB reactor within the standard deviations. Grey water sludge treatment with black water, therefore, does not deteriorate the effluent quality of the UASB reactor.

3.3. Excess Sludge Quality

In addition to increased energy recovery by grey water sludge treatment, soil application of black water sludge is a prerequisite for maximum carbon recovery. To investigate the influence of grey water sludge treatment on the UASB excess sludge quality, the heavy metal contents (mg/kgDW) of grey water sludge and the influent and excess sludge of the UASB reactors are determined and compared with the Dutch sludge reuse guidelines (Table 4). The heavy metal content of grey water sludge is higher compared to black water (BW-UASB reactor influent) with the exception of Cr, Hg and Ni, resulting in a higher heavy metal content of MIX-UASB reactor influent. The reuse of black water sludge is currently prohibited in the Netherlands, due to the elevated Cu and Zn concentrations. The excess sludge of the MIX-UASB reactor has a higher heavy metal content compared to the BW-UASB reactor, but the heavy metals are below the sludge reuse guidelines, with the exception of Cu, Zn and Ni. Grey water sludge contributes 36% (Cu), 32% (Zn) and 19% (Ni) to the heavy metal input in the UASB reactor on a solid matter basis. Since grey water sludge contributes only 23% to the input of solid matter in the reactor, grey water sludge addition increases the heavy metal concentration in the excess sludge. Furthermore, as heavy metals in black water are primarily human originated (feces and urine) and belong to the soil/food cycle [28], the introduction of external heavy metal sources by grey water sludge addition deteriorates the excess sludge quality and might therefore hinder its soil application.

Table 3. Removal efficiencies and characteristics of the influent and effluent of the UASB reactors at steady state and the characteristics of the grey water sludge added to the MIX-UASB reactor. TN, total nitrogen; TP, total phosphorus.

Parameter	Unit	BW-UASB		Removal (%)	GW sludge	MIX-UASB		Removal (%)
		Influent	Effluent			Influent	Effluent	
pH	–	8.1 (0.23)	7.8 (0.51)	–	7.2 (1.3)	8.1 (0.12)	7.3 (0.18)	–
COD _{total}	gCOD/L	11 (4.1)	1.1 (0.36)	90	15 (13)	12 (3.9)	1.3 (0.66)	88
COD _{suspended}	gCOD/L	6.4 (2.9)	0.23 (0.26)	96	15 (12)	8.5 (3.0)	0.25 (0.21)	96
COD _{colloidal}	gCOD/L	1.2 (0.63)	0.28 (0.13)	77	0.39 (0.37)	0.84 (0.42)	0.30 (0.20)	64
COD _{soluble}	gCOD/L	2.9 (0.94)	0.57 (0.075)	80	1.1 (0.68)	1.5 (0.43)	0.53 (0.19)	65
VFA	gCOD/L	0.99 (1.0)	0.020 (0.055)	–	0.85 (0.52)	0.25 (0.15)	0.039 (0.069)	–
TN	gN/L	1.2 (0.22)	1.1 (0.12)	8	0.46 (0.29)	1.3 (0.22)	1.1 (0.21)	15
TP	gP/L	0.19 (0.085)	0.090 (0.034)	53	0.12 (0.07)	0.28 (0.10)	0.14 (0.081)	50
BOD ₅	g/L	–	0.16 (0.030)	–	–	–	0.20 (0.050)	–

Note: () standard deviation; – not determined.

Table 4. Heavy metal content (mg/kgDW) of grey water sludge, influent and excess sludge of the UASB reactors and the Dutch sludge reuse guidelines.

Element	BW-UASB		GW Sludge	MIX-UASB		Dutch Sludge Reuse Guidelines ¹
	Influent	Excess Sludge		Influent	Excess Sludge	
As	0.30 (0.11)	0.75 (0.03)	0.65 (0.09)	0.57 (0.23)	0.88 (0.06)	15
Cd	0.17 (0.08)	0.76 (0.06)	0.29 (0.14)	0.30 (0.11)	0.70 (0.05)	1.3
Cr	77 (82)	39 (39)	57 (52)	35 (24)	62 (6.0)	75
Cu	95 (51)	220 (23)	172 (68)	161 (62)	331 (23)	75
Hg	0.01 (0.004)	0.006 (0.003)	0.004 (0.000)	0.004 (0.000)	0.01 (0.003)	0.75
Ni	46 (47)	25 (24)	34 (30)	20 (13)	33 (4)	30
Pb	1.4 (0.7)	4.4 (0.7)	17 (6.0)	15 (7.0)	12 (2.6)	100
Zn	284 (85)	821 (109)	441 (133)	431 (155)	1132 (68)	300

Note: (¹) BOOM [29]; () standard deviation.

3.4. Outlook

Anaerobic treatment of grey water sludge with black water increased the heavy metal content of the UASB reactor excess sludge and might therefore hinder its soil application. In the case where soil application of the sludge outweighs the benefits of increased energy production, another treatment system for grey water should be chosen. A sequencing batch reactor (SBR) studied by Hernández Leal *et al.* [30] for grey water treatment produces stabilized sludge with a lower volume due to a longer SRT compared to a bioflocculation unit and, therefore, benefits the disposal of the sludge. Furthermore, if grey water reuse is desired, SBR is the most favorable option, due to the high effluent quality [23]. For minimum energy consumption, an anaerobic step, such as the UASB reactor, followed by an aerobic step, such as a constructed wetland or an SBR, is recommended for grey water treatment [12]. Ultimately, the origin of heavy metals in grey water should be assessed to investigate the possibility of reducing the heavy metal content and to optimize the resource recovery from grey water.

4. Conclusions

Grey water sludge treatment with black water increased the energy recovery by 23% in the UASB reactor compared to black water treatment. The increase in the energy recovery can cover the increased heat demand of the UASB reactor and the electricity demand of the grey water bioflocculation system with a surplus of 0.7 kWh/cap/y electricity and 14 MJ/cap/y heat. However, grey water sludge introduced more heavy metals in the excess sludge of the UASB reactor and might therefore hinder its soil application.

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Author Contributions

Taina Tervahauta is the corresponding author and has been the main person responsible for the execution of the experimental work and the writing of the manuscript. Isaac M. Bryant has performed a significant part of the experimental work. Lucía Hernández Leal, Cees J. N. Buisman and Grietje Zeeman provided essential support in the experimental design and manuscript writing.

Appendix

Table A1 presents the sample composition and BMP in the batch experiments. The pH stays within the range of 7.5–8.3 during the experiments. The BMP of black water (61%) and grey water sludge (92%) are in accordance with previous studies [9,14,15].

Table A1. Sample composition and biochemical methane potential (BMP) in the batch experiments.

Parameter	Unit	Black Water	Grey Water Sludge	MIX (5 BW:1 GW-S)
COD _{total}	g/L	8.5	25	11
AS	mg/L	189	987	309
BMP	%	61 (10)	92 (0)	88 (3)

Note: () standard deviation.

Conflicts of Interest

The authors declare no conflict of interest.

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