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ORIGINAL ARTICLE

A case study for a cost-benefit-based, stepwise optimization of thermo-chemical WAS pre-treatment for anaerobic digestion

Magdalena Nagler[1] · **Peter Aichinger**[2,3] · **Martin Kuprian**[1,3] · **Thomas Pümpel**[1] · **Heribert Insam**[1] · **Christian Ebner**[1,3]

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Abstract Waste-activated sludge (WAS) may be considered a resource generated by wastewater treatment plants and used for biogas-generation but it requires pre-treatment (PT) for enhanced biogas-yields and reduced WAS disposal costs. To date, a number of studies on the optimization of such PT focused on improved biogas yields but neglected inferred energy and resource consumption. Here, we aimed to identify the most promising thermo-chemical PT-strategy in terms of net energy output and cost-efficiency by optimizing PT temperature and the amount and sort of the alkaline reagent used. We compared methane-potentials and disposal costs of untreated and treated WAS and conducted an annual cost-benefit calculation. We defined 70 °C and 0.04 M NaOH as ideal PT-conditions being both, low-energy demanding and efficient. Applying these conditions, enhanced biogas-yields and improved dewaterability led to reduced electricity and disposal costs of 22 and 27%, respectively, resulting in savings of approx. 28% of the yearly WAS-related expenditures of a wastewater treatment plant. Despite multiple benefits in running costs, the implementation of WAS-PT was not recommendable in the presented case study due to high investment costs.

Keywords Waste-activated sludge · Thermo-chemical pre-treatment · Cost-benefit analysis · Alkaline pre-treatment · Methane potential

Introduction

Waste-activated sludge (WAS) is formed at a consistent rate during the treatment of wastewater, representing around 30% (w/w) of the chemical oxygen demand (COD)-load of waste water treatment plants (WWTP) [1]. To minimise odor, volume, disposal costs and biological activity of the sludge and to harvest energy, WAS is often used at WWTP to produce biogas. This methane-rich gas is used to supply heat and electricity for the wastewater treatment process and buildings. Although the biodegradability of WAS by anaerobic digestion varies with certain characteristics such as COD:TOC ratio, carbohydrate, protein and lipid concentrations, the biogas yield is generally relatively low with 50–200 $\mathrm{L_{CH4}}$ $\mathrm{kg^{-1}_{volatile\ solids(VS)}}$ compared to 600–800 $\mathrm{L_{CH4}}$ $\mathrm{kg^{-1}_{VS}}$ for municipal organic waste [2–4]. Pre-treatment of this substrate, however, shows great advantages and may allow an immediate and distinct increase in methane yields [3].

Due to these low native energy yields but promising increases after PT, WAS is in fact one of the widest studied

✉ Magdalena Nagler
magdalena.nagler@uibk.ac.at

Peter Aichinger
aichinger@alps-gmbh.com

Martin Kuprian
kuprian@alps-gmbh.com

Thomas Pümpel
thomas.puempel@uibk.ac.at

Heribert Insam
heribert.insam@uibk.ac.at

Christian Ebner
c.ebner@avzirl.at

[1] Institute of Microbiology, University of Innsbruck, Technikerstr. 25d, 6020 Innsbruck, Austria

[2] Institute of Infrastructure, University of Innsbruck, Technikerstr. 25, 6020 Innsbruck, Austria

[3] AlpS-Center for Climate Change Adaptation, Grabenweg 68, 6020 Innsbruck, Austria

substrates in terms of pre-treatment (PT) strategies such as biological, thermal, microwave, mechanical, ultrasonic, pulsed electric fields, freeze/thaw, chemical and wet oxidation [5]. Of all these strategies, thermal and chemical PT seem to be the most efficient in terms of full-scale applicability, efficiency and economic profit [6–8]. Up to now, several studies have been conducted focusing on either single or combined PT using heat and alkaline reagents. Regarding the temperatures, ranges from 25 to 275 °C have been investigated, and for the alkaline reagent, several chemicals [KOH, $Ca(OH)_2$, NaOH] in different concentrations were tested [5, 9].

With thermal PT, microbial cell walls are broken down, releasing the intracellular, more soluble organic matter, increasing the resulting degradation efficiency but also the energy demand of the PT with increasing temperatures. Most studies suggest a range 160–180 °C for 30–60 min for optimum results [e.g. 10] (cf. Table 1). However, it has also been shown that temperatures of 175 °C and above may even reduce the bioconvertibility of WAS to methane due to the thermal degradation of soluble organics to undefined and to some extent insoluble, refractory compounds [11].

Alkaline PT, however, breaks down cell walls through saponification and leads to release of soluble COD and higher biodegradability of the substrate [5]. Its drawbacks are enhanced corrosion of the equipment and the need for a neutralization step if leading to a final pH >8 in order not to harm the microbial community and to inhibit the fermentation [12]. Furthermore, the surplus of alkalinity supports acidogenic reactions (nitrification) if process waters are returned to the activated sludge process [13].

Combined thermo-chemical PT is often conducted to merge the positive effects of both treatments. On the one hand, increased temperature was shown to counteract the negative effect of sodium on the WAS dewaterability, allowing NaOH to be applied as the most efficient PT-chemical [14–17]. On the other hand, the addition of NaOH allows the PT to be run at lower temperatures and to save heating energy.

In the last 35 years, a large range of temperatures as well as NaOH concentrations have been tested (cf. Table 1) and showed highly variable results due to differing conditions. Most of these studies, however, do not account for side effects like increased energy input that will decrease the net energy output or overall cost alteration, but focus on biogas production only. Therefore, usually the highest tested values have been declared to be the optimum temperatures or NaOH-concentrations. Regarding the reaction time, ranges from 10 min to 48 h have been applied. It has been shown that the efficiency increases with pre-treatment time, but that 60–71% of the 24 h-solubilisation of COD was achieved during the first 30 min of heating [18].

This study examines the optimization of the WAS PT, investigating the most appropriate combination effect of temperature and alkaline treatment and evaluating their effects by applying a calculation of the associated costs and benefits. The method of choice is not a full factorial design investigating the effects of each applied temperature with every possible concentration of alkaline reagent, as such data have already been gathered by others (cf. Table 1). In these former studies, the combined effects of thermo-alkaline treatment with NaOH have already been proven and an inhibitory effect can be excluded. Here, we rather aim to

Table 1 Studies focusing on combined thermo-chemical pretreatment (PT) on WAS using NaOH as chemical agent

References	PT range			Optimized conditions					
	T (°C)	NaOH (M)	Time	*T* (°C)	NaOH (M)	Parameter	Biogas prod. (L_N/kg_{VS})	CH_4 prod. (L_N/kg_{VS})	CH_4 content (%)
[11]	150–275	0.3	1 h	175		Methane production			
[15]	121	0–21 g/L	30 min	121	7 g/L	BG/methane production	194	130	70
[30]	25, 35, 55	0–0.09	**4**, 12 h	55	0.05	Methane production		310	72
[7]	130, 170	0.03, 0.065	1 h	170	0.03	BG production	228		
[31]	50, 60, 70, 80	pH 10, 11, 12	6–48 h	60	pH 12	% VS, % TS, BG production	862	569	65–69
[32]	70, 80, 90	1, 2, 3	10, **20**, 30 min	89	2.29	% disintegration			58.9
[6]	60, 75, 90	0, 0.1, 0.2	6 h	90/ 73.7	0.16/ 0.1	Sludge disintegration/ methane production		191	

Bold values indicate optimized conditions of the respective study proceed in consecutive steps, fixing one variable at a time conducting specific experiments and combining the results with knowledge from the literature, focusing on a preferably low energy- and cost-effort, in order to find the most efficient thermo-alkaline PT method for WAS with regard to net energy output and effective costs.

Materials and methods

A consecutive approach was chosen in order to identify the most efficient thermo-alkaline PT: (1) to deduce the most promising temperature to be combined with alkaline treatment, the influence of temperature was tested over a range 39–200 °C on several parameters indicating cell disruption. (2) The suitability of biomass ash, aluminate and NaOH as alkaline reagents in various concentrations was tested and the treatment yielding highest degrees of disintegration in combination with low energy input was defined as optimum PT. (3) For this most promising PT, the resulting WAS biological methane potential and dewaterabilities were determined in order to (4) finally conduct a cost-benefit calculation including all WAS-related costs for pre-treated and native WAS.

Waste-activated sludge

All experiments were performed with sludge of a conventional activated sludge process from the WWTP Zirl (Tyrol, Austria), a municipal WWTP treating wastewater from 61,000 population equivalents. The samples were taken after polymer dosage and mechanical excess sludge dewatering with a final content of total solids (TS) from 4 to 6% (w/w), volatile solids (VS) of 70% and a pH of 7.

Physico-chemical parameters

Total solids (TS) and volatile solids (VS) were both measured gravimetrically according to standard procedures (EN 12889, 15169) and the pH was determined using a pH-probe (744 pH Meter, Metrohm, Switzerland). All samples were measured in triplicates.

Temperature pre-treatments

Experiments were made in triplicates at temperatures of 39, 50, 70, 85, 100, 120, 150, 180 and 200 °C with a reaction time of 1 h, a time that had been found optimal by a number of studies [19]. Ambient pressure disintegration up to 100 °C was performed in 50 mL tubes heated in a water bath. To ensure a reaction time of at least 1 h at the given temperature, the core temperature of one sample was monitored. The high pressure disintegration from 120 to 200 °C was performed in a microwave heated pressure capsule (Speedwave, Berghof®) with the same sludge amount, heating and holding time. For the subsequent determination of disintegration parameters, the liquid and solid fractions were separated by two consecutive centrifugation steps at 3200×*g* and 37,000×*g* for 10 min each. The CODs after consecutive centrifugation was slightly higher than conventional CODs estimation by 0.45 µm filtration (<10%). However, this method was preferred to avoid intense filter plugging. The effects of the degree of disintegration were negligible (<1%).

Disintegration parameters

To determine the most suitable PT temperature, several parameters related to subsequent biogas production were measured to increase the reliability of the results:

- Dissolved COD (COD_s), increasing with cell disruption and disintegration of cell compartments and being positively correlated to the biodegradability and the resulting biogas-potential [5].
- Dissolved potassium (K), originating from broken microbial cells, as a possible parameter to measure the efficiency of cell disintegration [20].
- Dissolved organic carbon (DOC), normally formed during the first step of anaerobic digestion, the hydrolysis, but also released from broken cell walls after thermal treatment. It acts as a nutrient source for microorganisms and is supposed to correlate with the biogas potential of a substrate.

COD_S was measured photometrically in the supernatants after appropriate dilution with deionised water (Macherey–Nagel Nanocolor® CSB 1500). K was measured by atomic absorption spectrometry [21] after dilution with 1% CsCl as matrix modifier (Analytik Jena, ContrAA 700), and DOC was determined using an elemental analyser (Shimadzu, TOC-V CPN). For DOC and K, respective values for the dilution liquids were determined and subtracted from the results of the samples.

Finally, a highly disintegrated reference was generated by mixing the sludge 1:1 with 1 M NaOH and heating it to 90 °C for 10 min [22]. The sludge treated in such a manner was considered to have 100% and the native sludge 0% disintegration level and the corresponding COD data were called COD_{max} and COD_{S0}, respectively. Using these parameters, an additional value for the evaluation of the degree of disintegration (DD) [5] was calculated in order to compare the achieved data to literature data following Eq. (1)

$$DD_{COD}(\%) = \frac{(\mathrm{CODs} - \mathrm{CODs0})}{(\mathrm{CODmax} - \mathrm{CODs0})} \times 100, \tag{1}$$

Alkaline pre-treatments

After the evaluation of treatment temperatures, the most promising temperature was applied for all further experiments: In triplicates, 50 mL of WAS was mixed with a suitable amount of 1 M NaOH to achieve concentrations of 0.08, 0.04 and 0.02 mol L^{-1} and was immediately heated to 70 °C for 1 h in the water bath under continuous stirring. For the alkaline reagents ash and aluminate, one representative molarity was selected (0.04 M) to treat the sludge in the same manner. To do so, the alkalinity of both substrates was determined by titration of the diluted product with 0.5 M HCl to pH 4.3, and the results were used to produce a 1 M stock solution to be used for the experiments. The ash (bottom ash with a small amount of fly ash) was obtained from the biomass power plant Bioenergie Kufstein (Tyrol, Austria) and was sieved to 2–5 mm particle size prior to use, the aluminate was achieved from Donau Powerfloc flow. The pH of any chemically pretreated sludge was determined after reaching the final temperature using a pH-probe (744 pH Meter, Metrohm, Switzerland).

Methane potential and dewaterability of the digestate

The most promising combination of chemical and thermal pre-treatment regarding enhanced expected Biogaspotential due to DD_{COD} in combination with low energy input was used to determine the biogas potential in relation to an untreated WAS sample, using the Automated Methane Potential System (AMPTS; Bioprocess Control, Lund, Sweden) and following a standardised procedure [23]. Digested sludge from the WWTP Zirl was used as seeding sludge and allowed to degas at 37 °C for 7 days. In triplicates, negative controls with seeding sludge only, positive controls with microcrystalline cellulose (Alfa Aesar A17730) with a theoretical biogas potential of 740–750 L kg_{VS}^{-1} and mixtures of pre-treated and native samples with a ratio of $VS_{Sample}/VS_{Seeding\ sludge} = 0.5$ were anaerobically digested at 37 °C for 21 days.

TS, VS, pH, total proteins (DC Protein-Assay, Bio-Rad, Hercules, US) and the total COD were measured before and after the fermentation.

In order to estimate the dewaterability of the pretreated sludge in comparison to untreated sludge, two digestion runs were performed, one with untreated sludge, and the second one with pretreated sludge. 1 L batch reactors, containing 0.5 L of sludge were fed daily with pretreated or native substrates for 83 days (digester load: 2 kg_{VS} m^3 day^{-1}). Dewaterability was analyzed thermogravimetrically by the consulting company *Kläranlagen Beratung Kopp*, Lengede, Germany and expressed determining the value TR_A [24], a parameter defining the achievable TS of the sludge and the required amount of polyelectrolyte (PE) (ZETAG® 8160 BASF). This method was found to be the only suitable lab-scale method to predict actual dewatering results in full scale units [24].

Cost-benefit calculation

Applying a cost-benefit calculation, the advantages gained by the usage of the optimally pre-treated sludge were evaluated, including the actual costs for energy, NaOH, polyelectrolyte (PE) as well as digestate disposal costs and subtracting the benefit of the savings of electrical power deduced from biogas production. To account for heat energy, no additional costs were assumed for the pre-treatment of WAS, since heat at 70 °C is sufficiently available at the WWTP Zirl. The costs for NaOH were calculated assuming a prize of 350 € t^{-1}, digestate disposal was accounted for with 70 € t^{-1}, the cost for the polymeric flocking agent with 3 € kg^{-1} and the savings for electricity were calculated with 0.10 € kWh^{-1} (average purchase costs for major customers). For service and maintenance, personnel costs of 4 h per week were assumed and charged with 35 € h^{-1}. All costs and benefits were calculated for an annual balance with 10,000 t WAS (corresponding to approx. 40,000 population equivalents) and for a WAS with 5% TS and 70% VS.

Results and discussion

Temperature pre-treatment

DOC, COD and K all suggest similar disintegration trends, showing two maxima; a first peak at 70 °C (DOC and DOC) and 85 °C (K), followed by lower values at 120 °C and rising to maximum values at 200 °C (Fig. 1). Looking at the relative values, DOC and DD_{COD} suggest rather similar degrees of disintegration lying at 26 and 29% resp. for the 70 °C peak and rising up to 54 and 63% for samples treated at 200 °C. For K, the relative disintegration is about 62% at the 80 °C peak and practically the same (63%) at 200 °C.

The reduced disintegration efficiency at 100–120 °C is in accordance with Li and Noike [25], who measured the solubilisation ratio of VS after thermal WAS PT from 30 to 170 °C. Similarly, Kuglarz et al. [26], who measured dissolved COD after thermal PT from 20 to 100 °C, found stable but not increasing COD solubilisation rates as well as decreased ammonia and phosphate release from 70 to 100 °C. One possible explanation for this effect could be

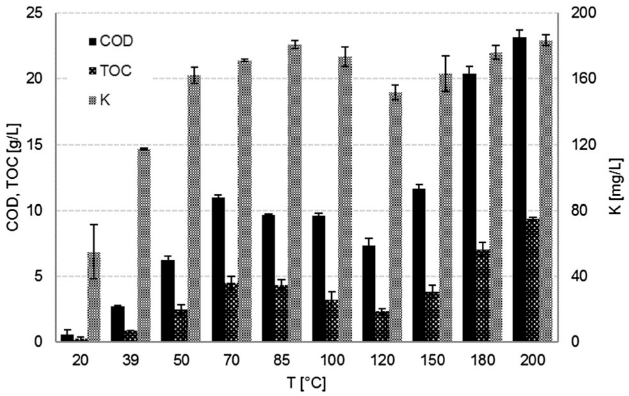

Fig. 1 Cell disintegration at various temperatures. Means and standard deviations of parameters indicating cell disruption at various disintegration temperatures (T). *COD* chemical oxygen demand, *K* dissolved potassium, *DOC* dissolved organic carbon

Maillard reactions, occurring between amino acids and reducing sugars at elevated temperatures over 80 °C and resulting in polymerization and thus diminished solubilisation of proteins [27].

Although two temperature optima for cell disintegration in the range 15–200 °C are visible, several reasons suggest the application of low temperatures around 70 °C for a thermal pre-treatment; (1) such a PT is energy saving due to the lower supply temperature level. Also, the heat from the combined heat and power unit of the WWTP can be used and the subsequent cooling step can be omitted, as the sludge can directly be mixed with cold primary sludge to a suitable temperature, counteracting the heat losses in the biogas reactor. (2) The PT can be performed at ambient pressure saving investment and operational costs. However, the level of disintegration is not yet fully exploited, leaving space for additional disintegration using an alkaline reagent.

This temperature is also supported by other studies, showing that it requires the lowest energy input to solubilize WAS [26] and stating that it is best in terms of VSS-reduction at a pH of 8 [28]. For all these reasons, a PT temperature of 70 °C was chosen and the following experiments were executed at this temperature.

Alkaline reagents and combined thermo-chemical PT

In this experiment, the thermal PT at 70 °C yielded a DD_{COD} of 34% compared to the native WAS. This DD_{COD} was increased by the addition of equal alkalinity as ash (38%), NaOH (46%) and aluminate (47%). While NaOH is known to work well, ash and aluminate have not been tested as alkaline reagents for sludge PT and would have the advantage to be cheap industrial wastes.

When compared to the highest achieved value of the aluminate-treatment, the DD_{COD} of the thermal and the ash treatments where found to be significantly lower ($p < 0.001$, −11.6 and −9.2% resp.), while the NaOH-treatment was not significantly different (Fig. 2a). The pH, however, was lowest after the treatment without alkaline reagent (6.82) and increased with the addition of NaOH (7.85), ash (8.45) and aluminate (9.02). We determined NaOH as the optimal alkaline reagent due to the following reasons: if compared to aluminate, it shows the same degradation efficiency but has stable costs that are not subjected to the waste market. Furthermore, the utilisation requisites of waste aluminate are not yet clarified and if the waste product is used, it has a variable composition. Ash is less effective with regard to NaOH, although it has been found to act beneficial to methane concentrations and hydrolytic efficiency in cattle slurry-based biogas plants [29]. However, the here required amounts to reach the desired alkalinity of 0.04 M were rather high and would result in 400 t to be sieved and amended annually. Furthermore, the sedimentation process in the sludge was impacted with increased amendment of ash, lowering the overall economic feasibility of this treatment. Having in mind that additionally, a final pH >8 would probably require a neutralization step of the pretreated sludge [cf. 12] increasing the costs of the pre-treatment, these results suggest the use of NaOH as preferred alkaline reagent.

Compared to the thermally at 70 °C pre-treated sludge (25% DD_{COD}), the DD_{COD} increased by 24% using 0.02 M NaOH (49% DD_{COD}), by 42% using 0.04 M NaOH (67% DD_{COD}) and by 50% using 0.08 M NaOH (75% DD_{COD} Fig. 2b), showing a non-linear relationship of DD_{COD} and NaOH-concentration and suggesting the use of 0.04 M NaOH to be the most efficient in terms of chemical costs vs degradation capacity and being consistent with the optimum concentrations defined in other studies [7, 30].

Dewaterability and methane potential

Applying the optimized PT-conditions 70 °C and 0.04 M NaOH, the dewaterability of the WAS increased from 27.0% TR_A for the native sludge to 32.6% TR_A for the treated sludge, reducing the disposal costs for the remaining WAS substantially. The necessary amounts for the treatment with PE increased from 12.7 kg t TS^{-1} for the native WAS to 16 kg t TS^{-1} for the treated WAS.

With the optimized PT-strategy 70 °C and 0.04 M NaOH, the biogas potential test resulted in increased methane production of 213 $L_N\ kg_{VS}^{-1}$ compared to 174 $L_N\ kg_{VS}^{-1}$ for the native sludge (Fig. 3), comparable to the production rates observed elsewhere (cf. Table 1). This corresponds to an increase of 22.3% in methane yield. The activity of the microorganisms could be confirmed by their

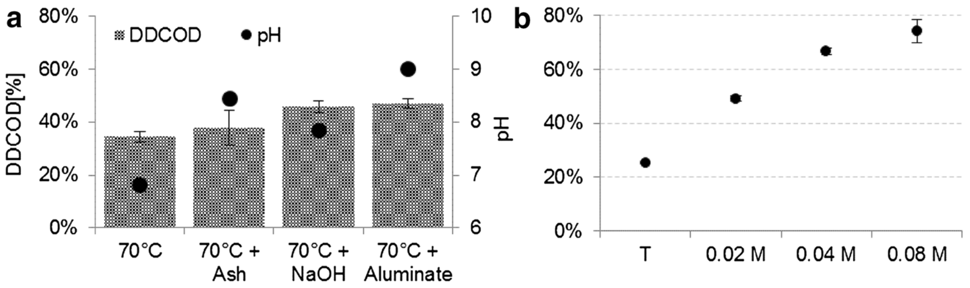

Fig. 2 Cell disintegration with various alkaline reagents and molarities. **a** Degrees of disintegration (DD) calculated using COD for thermally pretreated WAS (70 °C) and WAS treated with an additional alkaline reagent (ash, NaOH and aluminate) at an alkalinity of 0.04 M each. The secondary axis shows the corresponding pH of the sludge after treatment. **b** DD_{COD} achieved with different molarities of NaOH

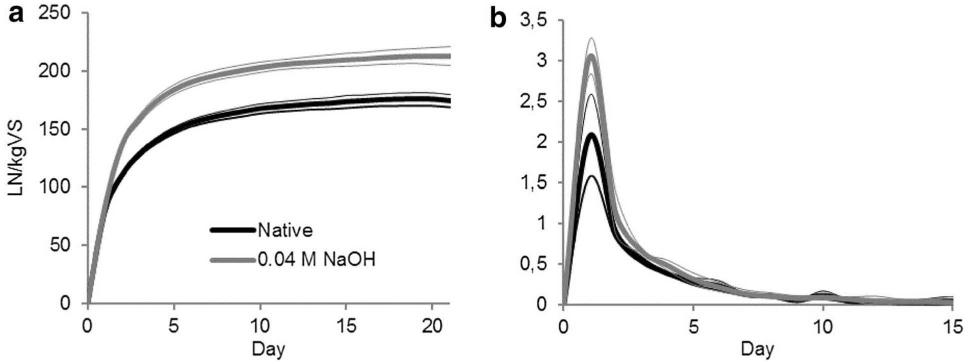

Fig. 3 Methane production of the optimally pre-treated vs native WAS. **a** Accumulated and **b** daily methane production of the native WAS and the WAS pretreated with 0.04 M NaOH at 70 °C. *Bold lines* represent means ($n = 4$), *narrow lines* the corresponding standard deviation

Table 2 Cost-benefit calculation for the additional costs and benefits generated by the pretreatment of WAS and expressed as €/a

	Native	Treated	Unit	% change
Energy				
Methane potential	174	213	$m_N^3\ t_{VS}^{-1}$	22
Electricity ($\eta = 35\%$)	213	260	MWh a^{-1}	22
Heat ($\eta = 40\%$)	243	297	MWh a^{-1}	22
Degradation efficiency				
Regular degradation (%)	50	50	(w/w)	
Additional degradation (%)	0	11	(w/w)	
TR_A (%)	27	33	(w/w)	21
Affected parameters				
Electricity generation	213	260	MWh a^{-1}	22
Disposal	1204	878	t a^{-1}	−27
NaOH	0	16	t a^{-1}	
Polymer	4128	4580	kg a^{-1}	11
Calculated effective costs				
Costs/return electricity	−21,300	−26,014	€ a^{-1}	22
Disposal	84,259	61,468	€ a^{-1}	−27
NaOH	0	5600	€ a^{-1}	
Polymer	12,383	13,741	€ a^{-1}	11
Personnel	0	4400	€ a^{-1}	
Overall costs	75,342	59,195	€ a^{-1}	−21

methane production of 362 $L_N\ kg_{VS}^{-1}$ for microcrystalline cellulose, representing 87% of the theoretical methane potential [23]. These results also confirm that the resulting increased pH of the pre-treated sludge was not inhibiting the microbial consortium. Regarding the physico-chemical parameters, TS, total COD and total proteins where all degraded to a higher extend in the treated substrate (5 vs 13% reduction in TS, 15 vs 18% reduction of total COD and 10 vs 14% reduction in total proteins for native and treated sludge, resp.), while VS showed a similar reduction for treated and native sludge samples (4.2 vs 3.8% reduction for native and treated sludge, resp.).

Cost-benefit calculation

The cost-benefit calculation shows that the WWTP has overall yearly WAS-related costs of 75,342 € a^{-1} and that these costs drop by 21% to 59,195 € a^{-1} with the optimised WAS PT (cf. Table 2). While the costs for heating can totally be retrieved from other WWTP processes by using excessive heat, the most important costs generated by the PT are the chemicals used for the alkaline PT itself (NaOH; 5600 € a^{-1}), the personnel costs (4400 € a^{-1}) and the increased polymer demand for the flocculation of the pretreated WAS (1358 € a^{-1} increase compared to native sludge). Nevertheless, the increased savings in electrical power resulting from increased biogas production (4714 € a^{-1} more savings for treated WAS, −22%) and especially the reduced disposal costs (22,791 € a^{-1} less for treated WAS; −27%) lead to the overall positive effect of the PT (cf. Table 2). With the currently chosen cost parameters, such a PT would lead to total savings of 16,147 € a^{-1}. Not included in the cost-benefit calculation is the investment for the construction of a PT-facility, an investment that would have to be offset by the money saved yearly in less than 15 years, the regular lifetime of such a facility. If estimated with 360,000 €, it will be amortised within 22 years with the here-chosen parameters, causing the PT not to be beneficial enough to be implemented. If, however, the WWTP treats greater amounts of WAS, the PT will turn to be beneficial as soon as these amounts rise above 14,000 t (56,000 PE). Additionally, the overall savings could be enlarged by omitting an often necessary pre-heating of the native sludge prior to the introduction into the digester and by an additional compensation for the thermal transmission losses achieved with the higher temperatures of the pre-treated sludge.

Conclusion

The here-optimized WAS-PT requires a 1 h thermo-chemical treatment at 70 °C with 0.04 M NaOH. Applying these conditions, maximum PT efficiency in terms of sludge disintegration can be achieved in combination with low heat and chemical-related costs. In addition to an increased methane and biogas yield, such a PT leads to enhanced sludge degradation during anaerobic digestion and a higher dewaterability of the digested sludge, decreasing amount of waste to be disposed and resulting in savings of approx. 21% of the yearly WAS-related expenditures of a WWTP.

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