Effects of post anaerobic digestion thermal hydrolysis on dewaterability and moisture distribution in digestates

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ABSTRACT

Organic waste fractions such as sewage sludge, food waste and manure can be stabilized by anaerobic digestion (AD) to produce renewable energy in the form of biogas. Following AD the digested solid fraction (digestate) is usually dewatered to reduce the volume before transportation. Post-AD treatments such as the Post-AD thermal hydrolysis process (Post-AD THP) have been developed to improve the dewatering, but the mode of action is not well understood. In this study, samples from 32 commercial full-scale plants were used to assess the impact of Post-AD THP on a broad range of raw materials. Maximum dewatered cake solids after Post-AD THP was predicted by thermogravimetric analysis (TGA). Post-AD THP changed the moisture distribution of the samples by increasing the free water fraction. A consistent improvement in predicted dewatered cake solids was achieved across the 32 samples tested, on average increasing the dry solids concentration by 87%. A full-scale trial showed that dewatering Post-AD THP digestate at 80 °C improved dewatered cake solids above the predictions by TGA at 35 °C. In conclusion, dewatered cake solids were significantly improved by Post-AD THP, reducing the volume of dewatered cake for disposal.

Key words | anaerobic digestion, biogas, dewatering, moisture distribution, thermal hydrolysis

INTRODUCTION

Anaerobic digestion (AD) is a common method to reduce and stabilize sewage sludge and organic waste (Aguilar et al. 2017). The process results in two end-products: biogas and digestate. The biogas can be directly used at the plant as an energy source in combined heat and power (CHP) engines. At some plants the biogas is upgraded to vehicle fuel or injected to the gas grid (Mills et al. 2014). The digestate is usually dewatered to separate the solids from water,
producing a cake and a liquid fraction. Minimizing the water content in the dewatered cake reduces the total cost of dewatering and cake handling that can represent 30–50% of a plant’s operational budget (Mikkelsen & Keiding 2002). Efficient dewatering will depend on sludge physicochemical properties, polymer type and dose for particle aggregation and type of dewatering device (Novak 2006). However, pre-treatment methods altering the sludge structure are necessary to significantly improve dewaterability (Neyens & Baeyens 2003).

Pre- or post-treatment processes such as heat treatment, chemical treatment, mechanical disintegration, freezing and thawing and biological hydrolysis with or without enzyme addition have been explored to intensify the digestion process and improve dewatering efficiency (Neyens & Baeyens 2003). The thermal hydrolysis process (THP) is an established hydrothermal treatment process traditionally applied prior to the AD process (Pre-AD THP) with more than 75 facilities in operation or in design worldwide (Barber 2016). Pre-AD THP typically operates at 165 °C sterilizing the biomass and enabling increased digester loading rates through reduced sludge viscosity (Barber 2016). In addition, improved biogas production and digestate dewaterability have been reported (Barber 2016; Svennevik et al. 2019). The positive influence on dewatering by lowering viscosity has been studied by several authors, and could explain the positive influence of THP on dewatering (Klinksieg et al. 2007; Miryahyaei et al. 2019). Poor dewaterability of biological sludge has been attributed to the strong water binding capacities of microbial extracellular polymeric substances (EPS) which could represent up to 80% of the biomass (Neyens et al. 2004; Christensen et al. 2015). Application of sludge treatment such as thermal hydrolysis has been shown to degrade the EPS, hence reducing the water retention properties of sludge (Neyens et al. 2004). Therefore, the reduction of viscosity caused by THP could be associated with changes in the moisture distribution.

Although Pre-AD THP is widely applied, the THP was originally developed as a dewatering aid directly in front of the dewatering device (Barber 2016). Recent full-scale and laboratory studies on the application of the THP after AD (Post-AD THP) have shown encouraging results with large improvements in dewatering (Kolovos et al. 2016; Svensson et al. 2018). More than 60% reduction in dewatered cake for disposal has been reported for a full-scale Post-AD THP plant (Kolovos et al. 2016) and similar results were found in the laboratory for sewage sludge digestate (Svensson et al. 2018). However, Post-AD THP on fiber-rich source separated food waste (SSFW) digestate resulted in only 26% wet cake reduction (Svensson et al. 2018) which could indicate that the effect may be substrate dependent.

Due to the many full-scale Pre-AD THP installations a wide range of samples have been analysed, and laboratory trials have been performed to assess the effect of this configuration on dewaterability (Neyens et al. 2004; Barber 2016; Svennevik et al. 2019). Solids characteristics have been found to influence the dewatered cake solids also after Pre-AD THP (Skinner et al. 2015; Barber 2016; Svennevik et al. 2019). Thus, large variations in digestate characteristics can be expected between different full-scale plants. To date, only one full-scale plant is using Post-AD THP making it challenging to assess the effect of this process on dewaterability of different digestates in full-scale. In order to predict the effect of this technology in practice a better understanding of the mechanisms influencing the dewaterability after Post-AD THP on a wide range of raw materials is needed. The effect of Post-AD THP on the moisture distribution has to our knowledge so far not been studied but may explain the superior dewatering performance of this technology.

Reported improvements in dry solids concentration of dewatered cake due to industrial application of Post-AD THP on sewage sludge surpassed the effect of Pre-AD THP (Gerstner et al. 2017). This improvement make Post-AD THP a promising technology for plants with high cake disposal costs, as cake disposal savings are becoming an increasingly important economic advantage of installing THP in novel wastewater treatment plants (Taboada-Santos et al. 2019). In addition, significant improvements in dewatering could affect the design and footprint of new plants. The area needed for storage of dewatered cake can be reduced, less trucks are needed for cake disposal and alternative cake disposal options could emerge due to the higher dryness of the dewatered cake. However, in order to implement these factors in the planning of new plants, more information is needed on the expected dry solids concentration of dewatered cake after Post-AD THP.

In addition to the effect of THP treatment, increasing the digestate temperature has been reported to have a positive effect on both sludge viscosity and dewatering (Klinksieg et al. 2007). However, the highest temperature studied by Klinksieg et al. (2007) was 55 °C after thermophilic digestion while the Post-AD THP dewaters at 80 °C. Hence, more research is needed to understand the effect of temperature on dewatering, particularly for the temperature range associated with Post-AD THP dewatering.

To better understand the mechanisms and universality of the Post-AD THP technology a wide range of biomass
samples were analyzed in this study focusing on the dry solids concentration of dewatered cake. The aims were to: (1) study the effect of Post-AD THP on the free water and predicted dry solids concentration of dewatered cake, and (2) study the effect of high temperature dewatering on the dry solids concentration of dewatered cake.

**MATERIALS AND METHODS**

**Samples**

A wide range of digestates and dewatered digestates were collected from a total of 32 plants in Europe, Asia and Oceania to study the effect of Post-AD THP on predicted cake solids (Table 1). Dewatered cake solids or predicted cake solids by thermogravimetric analysis (TGA) from Plants A-H, J and I-U have earlier been published by Svennevik et al. (2019).

All samples were shipped to the Norwegian University of Life Sciences in Norway and stored at 4 °C until analyzed.

**Thermal hydrolysis treatment**

To assess the impact of Post-AD THP two THP pilots were used. Dewatered digestate from Plants A-H (Table 1) was treated in a THP pilot at Reading Sewage Treatment Works, UK, previously described by Shana (2015). Dewatered digestates from Plant I – AF were treated with the Cambi Thermal Hydrolysis pilot (Cambi Group AS, Norway) located at the Norwegian University of Life Sciences (Horn et al. 2011). Both units were operated similarly and digestates were hydrothermally treated by steam injection to 6 barg for 45 minutes followed by direct steam explosion. The samples were stored at 4 °C until analyzed.

**Thermogravimetric analysis**

TGA was used to determine the free water content in accordance to Kopp & Dichtl (2001b) with minor modifications described by Svensson et al. (2018) and Svennevik et al. (2019). In brief; 100 mg samples were dried at 35 °C in a Netzsch Simultaneous Thermogravimetry-Differential Thermal Analysis/Differential scanning calorimetry (TG-DTA/DSC) Apparatus STA 449 F1 Jupiter® with a constant nitrogen flow of 20 mL/min. The drying curves (see Supplementary Figures S1–S32) were analyzed as described by Svennevik et al. (2019) to identify the transition point between free and interstitial water. Calibration was done with monodisperse silica particles of diameters 1.86 μm, 4.08 μm and 7.75 μm (Cospheric LCC, USA). Predicted dewaterability measured five times on the same sample was 40.6 ± 0.7% DS.
The maximum dry solids concentration of dewatered cake can be predicted based on the assumption that the free water measured by the TGA can be removed in full-scale centrifugation (Kopp & Dichtl 2001b). The prediction method has shown good results when compared to full-scale dewatered cakes (Kopp & Dichtl 2001b; Svennevik et al. 2019).

Low-field nuclear magnetic resonance

Low-field nuclear magnetic resonance (LFNMR) was used to determine bound water and water diffusion rates (WDR).

Bound water

Bound water in digestates and Post-AD THP digestates from Plants A-H were determined as described by Beck et al. (2018) and Svennevik et al. (2019). In brief: five mL of sample were analyzed by a Bruker mq20 minispec with a 0.47 T permanent magnet (Bruker, Billerica, MA, USA). The temperature was controlled at 22 °C with a BVT 3,000 nitrogen temperature control unit (Bruker, Billerica, MA, USA). Spin-spin relaxation time (T2 relaxation time) was measured by using the Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence (Carr & Purcell 1954; Meiboom & Gill 1958). The bound water was defined by the peak with the shortest relaxation time and calculated in relation to the total peak areas.

Water diffusion rate

WDR were measured on a 21 MHz (0.5 T) LFNMR spectrometer supplied by Anvendt Teknologi AS. WDR was used to investigate the effect of temperature (80 °C, 55 °C) on Post-AD THP digestates from Plants A, B, E-G and K. The instrumentation produces pulsed magnetic field gradients up to 400 G/cm for diffusion and one-dimensional image experiments. Three mL of sample were filled in LFNMR sample tubes of 18 mm diameter and adjusted to either 35 °C or 80 °C by heated air for 10 minutes. CPMG was used to measure T2 relaxation time (Carr & Purcell 1954; Meiboom & Gill 1958) and a set of diffusion measurements at different observation times (Sørland 2014). To investigate the repeatability of the water diffusion rate measurement, one sample was split into three and measured in triplicate yielding an average water diffusion rate of 2.7 ± 0.1 e⁻⁹ m²/s.

Moisture distribution

Three main water fractions were used in this study: free water, interstitial water and bound water (Kopp & Dichtl 2001b). The moisture distribution in digestates and Post-AD THP digestates from Plant A-H were determined as described by Svennevik et al. (2019). In brief, free water was determined by TGA and bound water by LFNMR. Interstitial water was quantified by subtracting the amount of free water from bound water. All samples were normalized to 5% DS by mathematically adjusting the free water content, to allow comparison between different samples. The moisture distribution of digestates in Plants A-H has earlier been published by Svennevik et al. (2019).

CENTRIFUGATION

Laboratory centrifugation

To study the effect of temperature (20 °C and 80 °C) on dewatering of Post-AD THP digestate a Beckman Model J2-MC Centrifuge with a JS-7.5 rotor was used for centrifugation. The samples were centrifuged at 1889 G for 30 minutes. Samples were stabilized at room temperature or heated in a heating cabinet 80 °C prior to centrifugation. After centrifugation the supernatant was decanted for soluble chemical oxygen demand (COD) analysis and the remaining pellets were analyzed for dry solids.

Full-scale centrifugation

The full-scale trial was done at Plant K with a mobile centrifuge from GEA Westfalia. Digestate at 80 °C was provided from the full-scale installation. The linear and high cationic charged polymer Zetag 9,118 (BASF, Germany) at a concentration of 0.2% active substance (AS) was used. Polymer dose is reported as kg AS/ton DS. The experiment used a minimal polymer dose of 12 kg AS/ton DS as this was needed to obtain good floc-formation.

Characterization analysis

The dry solids (DS) and volatile solids (VS) concentrations were measured gravimetrically by drying a sample at 105 °C to constant weight and subsequent combustion at 550 °C. Standard deviation represents three measurements on the same sample.
Soluble COD was measured after filtration at 0.45 µm with Merck Spectroquant® COD Cell Test. Standard deviation represents three measurements on the same sample.

ADF was analyzed according to manufacturer’s recommendations using an Ankom200 Fiber Analyzer (ANKOM Technology, Macedon, New York, USA) with F58 filter bags.

**Statistical analysis**

Single factor analysis of variance (ANOVA) was performed in Microsoft Excel to assess if two data sets were significantly different at a significance level of 0.05.

**RESULTS AND DISCUSSION**

**The effect of Post-AD THP on free water and predicted cake solids**

A wide range of digestates from 32 commercial full-scale plants with dewatered cake solids ranging from 19–35% DS were used to test the universality of Post-AD THP. Plants C, D, F and G were sampled for Post-AD THP testing twice, one year apart. There was no significant difference in predicted cake solids by TGA for the 2015 (data not shown) and 2016 (used in Figure 1) samples demonstrating good reproducibility of sampling procedure, treatment and analysis. The dry solids concentration of original dewatered cakes and predictions by TGA for Plants A-H, J and L-U have earlier been published by Svennevik et al. (2019). Predicted dewatered cake solids by TGA of Post-AD THP digestates were compared to the cake solids of original dewatered cake with open symbols representing digestates from SSFW (Figure 1).

After Post-AD THP a significant increase \( (p < 0.001) \) in predicted cake solids was found compared to the original dewatered cake solids (Figure 1). Regardless of the original cake dryness or any use of pre-AD treatment the predicted cake solids were on average increased by 87% by application of Post-AD THP at 165 °C for 45 minutes.

Solids characteristics such as VS content (Kopp & Dichtl 2001b; Skinner et al. 2015; Yu et al. 2017), carbon to nitrogen ratio (C/N) (Nicholson et al. 2018) or the combined factor CNash (Svennevik et al. 2019) has recently been used to explain the large span in dewaterability of sludges of different origins. VS content of original dewatered cake solids did not seem to significantly affect dewaterability for our broad set of samples (Supplementary Figure S33). However, Figure 1 show that both sludge type and thermal pre-treatment history impact the predicted dewatered cake solids after Post-AD THP. The consistent increase in predicted cake solids across the sample set could indicate that Post-AD THP uniformly influenced the different digestates. This could be attributed to the break-down of the porous network structure (Zhang et al. 2018), change in rheology (Klinksiieg et al. 2007; Stickland 2015; Barber 2016) and solubilization of the structural integrity of EPS (Neyens et al. 2004) resulting in the release of free water and improved dewaterability.

Samples from Plant P, T and U contained SSFW, showing an average improvement of 77 ± 31%. This is substantially higher than the result from Svensson et al. (2018) where a 35% improvement was found after Post-AD THP of SSFW digestate. This could be due to the fiber content in the digestates, as Plant P, T and U had a lower fiber content than the digestate tested by Svensson et al. (2018) (data not shown).

To better understand the reason for the observed change in free water (Figure 1), eight digestates (Plants A-H) were selected for moisture distribution analysis before and after Post-AD THP treatment. The digestates had a large span in primary sludge to waste activated sludge ratios, and different thermal pre-treatments or no pre-treatment. A significant increase in free water \( (p < 0.001) \) and reduction in interstitial water \( (p < 0.001) \) were observed after Post-AD THP (Figure 2). However, no significant difference in bound water was measured (Figure 2).

The increase in free water was almost equal to the reduction in interstitial water, jointly accounting for 99 wt.-% of the change in moisture distribution (Figure 2; data in Supplementary Table S1). Kopp & Dichtl (2001b) suggested
that capillary forces between the sludge flocs bind the interstitial water to the sludge. We hypothesize that the change in interstitial and free water can be attributed to the reduction of these capillary forces restricting the interstitial water fraction from behaving like free water. This can potentially be due to the degradation of EPS as THP has been shown to break down the water holding capacity of EPS (Neyens et al. 2007). Since the free water has been linearly correlated to the dry solids concentration of dewatered cake in full-scale (Kopp & Dichtl 2004), the change in moisture distribution is probably the main reason for the improved dewaterability after Post-AD THP reported by Kolovos et al. (2016) and Svensson et al. (2018).

In addition to differences in types of raw materials and any use of pre-treatment, the AD process is typically operated at either mesophilic (MAD) or thermophilic (TAD) temperatures. The influence of these two configurations on predicted cake solids after Post-AD THP dewatering was investigated using samples from Plant X, which ran these processes in parallel. The same predicted dry solids concentration in dewatered cake by TGA was found for both digestates (38% DS, data not shown).

The results provide novel information about the effect of Post-AD THP on an extensive data set, showing the universality of the technology to improve the concentration of solids in the dewatered cake. In addition, the effect of high temperature dewatering could promote further water extraction.

High temperature dewatering

The effect of dewatering at 80 °C compared to 20 or 35 °C was investigated in the laboratory to see if more water could be removed.

Laboratory trials

11 Post-AD THP digestates were centrifuged in the laboratory at 20 °C and 80 °C to study the effect of increased temperature on water extraction (Figure 3).

The experiment demonstrated a significant average increase of 22 ± 3% (p < 0.01) in water extraction from the pellet when increasing the digestate temperature from 20 °C to 80 °C. Using samples between 5 and 55 °C Klinksieg et al. (2007) developed a rheological model to predict dewaterability at elevated temperatures. Assuming their equation is valid up to 80 °C, an increase in cake solids of 25% could be expected. Hence, our result (22%) agrees well with the model developed by Klinksieg et al. (2007). Several factors may explain this increase.

Increasing the temperature up to 70 °C has been linked to the destruction of vicinal (surface) water (Vesilind 1994). If surface water decreases at 70 °C interstitial water that has a lower binding energy than surface water (Kopp & Dichtl 2004a) can to some extent be converted to free water. Based on this, our hypothesis is that when dewatering at 80 °C the binding forces previously restricting interstitial and surface water from behaving like free water are weakened and the dry solids concentration of the pellet was therefore increased.

Moreover, the increase in free water may be linked to the structural integrity of lipids or collagenous compounds at different temperatures. Lipids in aqueous media have higher water diffusion coefficients and solubility at higher temperatures (Chipasa & Međrzycka 2006). Thus, when solubilized at 80 °C this could potentially release some of the interstitial water to the free water fraction. The soluble COD of the supernatants obtained from centrifugation at 20 °C and 80 °C was measured, showing significantly
higher concentrations of soluble COD in the 80 °C supernatant (p = 0.02) (Figure 4) supporting this hypothesis.

In addition to the effect on structural integrity of lipids or collagenous compounds trapping water in the sludge matrix, water will also have a lower viscosity at 80 °C than 20 °C (Korson et al. 1969), which may also explain the positive influence of temperature on dewatering reported by Klinkseig et al. (2007). A linear relationship between increasing concentration of soluble COD and reduction in sludge viscosity due to thermal treatment up to 80 °C was found by Farno et al. (2015), linking the results from Figures 3 and 4 as reduced viscosity has been shown to improve dewaterability (Barber 2016).

Water viscosity in biomaterials can be related to its coefficient of permeability and hence the rate of thermal dewatering (Clayton et al. 2006). LFNMR can provide information on the mobility of water at different temperatures by measuring the water diffusion rate (WDR) in the digestate. The effect of temperature on the water diffusion rate in six Post-AD THP digestates was therefore measured by LFNMR (Figure 5). The lowest stable temperature with the current set-up was obtained at 35 °C (Figure 5). The lowest stable temperature with the current set-up was obtained at 35 °C and the WDRs at this temperature were compared to WDRs at 80 °C (Figure 5).

The WDRs at 35 °C and 80 °C were significantly different (p < 0.001) with 98 ± 14% higher WDRs at 80 °C compared to 35 °C. Korson et al. (1969) found that water had a 103% higher viscosity at 35 °C compared to 80 °C. The Post-AD THP digestates tested (Figure 5) consist of a large fraction of water (93 ± 3% water, data not shown), hence the reduced viscosity of this water could potentially improve the water release rate during dewatering. This is in line with findings of Clayton et al. (2006) showing that water removal from biomaterials in the initial thermal dewatering stage of dewatering by mechanical thermal expression was promoted when increasing the temperature up to 90 °C. This can positively influence full-scale dewatering since the time in the centrifuge is limited, making the rate of water release important (Kopp & Dichtl 2001b).

Full-scale centrifuge dewatering at 20–35 °C has been correlated to the fraction of free water measured by TGA at 35 °C (Kopp & Dichtl 2001b). This study indicates that this can be further improved by dewatering at 80 °C. A full-scale trial was therefore initiated to investigate if higher cake solids could be achieved than predicted by TGA and if so, at what polymer dose.

### Full-scale dewatering

The total cost of the full-scale dewatering process will depend on two factors: the polymer dose, as this product can be expensive and achieved concentration of dry solids in the dewatered cake, as this is directly related to the mass of cake for disposal. Full-scale Post-AD THP digestate (Plant K) with various amounts of polymer was used to study full-scale dewaterability at 80 °C (Figure 6).

Dewatered cake solids was predicted by TGA at 35 °C to be 39% DS, assuming optimal polymer conditioning before centrifuge dewatering. The prediction by TGA was achieved after centrifugation with a polymer dose of 12 kg active substance (AS)/ton DS when operating at 80 °C (Figure 6). This was the same polymer dose previously used with a Pre-AD THP configuration at the same plant.

The prediction of TGA would normally be the upper limit when dewatering at 35 °C with a centrifuge, and further polymer dosage would not increase the concentration of dry solids in the dewatered cake as reported by Kopp & Dichtl (2001b). However, in this study, increasing the polymer dose further increased the cake solids up to...
47.5% DS using 24 kg AS/tDS (Figure 6). We hypothesize that the amount of free water has increased at 80 °C compared to the predicted amount at 35 °C, hence increasing the maximum dewatered cake solids achievable in the centrifuge. To achieve the new maximum dewatered cake solids, we speculate that more polymer is needed to build denser and stronger flocs able to withstand the shear and centrifugal forces during dewatering in order to release the free water. The dewatered cake at 47.5% DS corresponds to a 21% increase in cake dryness compared to TGA predictions, supporting the positive effect of higher temperatures on the dewatered cake solids.

**RESEARCH IMPLICATIONS**

This study has shown that predicted cake solids after Post-AD THP dewatering from 32 commercial full-scale plants depended on the original dewaterability, but a consistent increase was found. The cost of dewatering and cake handling can have a large influence on the operational budget, hence the ability to predict the expected dry solids concentration of dewatered cake is important. Consequently, the improvement of 87% across the broad sample range studied will provide important information to support cost/benefit analysis prior to investments in plant upgrades or planning of new plants. A significant reduction in dewatered cake will reduce the needed cake storage area and the amount of trucks needed for disposal transportation, both factors that may influence the design of new plants. In addition, new opportunities for cake disposal or energy recovery may arise due to the higher dryness of the dewatered cake (Mills et al. 2014).

Dewatering at 80 °C showed further potential for improvement in cake solids when increasing the polymer dose compared to the predictions by TGA for conventional dewatering at 35 °C. The proposed mechanisms are a change in moisture distribution, the solubilization of lipids and collagenous compounds and the increased diffusion rates of water. The practical implication of this is a balance between the increased cost of polymer at higher dose compared to the reduced cost of transport and processing of the dewatered Post-AD THP digestate. These are important factors to include in the operational budget, and the cost of polymer must be balanced with the cost of cake handling.

**CONCLUSIONS**

In this study, novel insights across a broad range of raw materials were found on the impact of Post-AD THP on dewatering. Post-AD THP changed the moisture distribution of digestates by releasing the interstitial water into the free water fraction. An average cake solids increase of 87% after Post-AD THP was found. Furthermore, dewatering at 80 °C increased the amount of water extractable by a centrifuge above the predictions by TGA at 35 °C.

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**SUPPLEMENTARY DATA**

The Supplementary Data for this paper is available online at http://dx.doi.org/10.2166/wst.2019.379.

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