Water Research 158 (2019) 350–358

Contents lists available at ScienceDirect

Water Research

journal homepage: www.elsevier.com/locate/watres

CNash - A novel parameter predicting cake solids of dewatered digestates

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ARTICLE INFO

Article history:
Received 9 February 2019
Received in revised form
6 April 2019
Accepted 15 April 2019
Available online 23 April 2019

Keywords:
Anaerobic digestion
Ash
Biogas
C/N
Dewatering
Thermal hydrolysis

ABSTRACT

Efficient digestate dewatering is crucial to reduce the volume and transportation cost of solid residues from anaerobic digestion (AD) plants. Large variations in dewatered cake solids have been reported and predictive models are therefore important in design and operation of such plants. However, current predictive models lack validation across several digestion substrates, pre-treatments and full-scale plants. In this study, we showed that thermogravimetric analysis is a reliable prediction model for dewatered cake solids using digestates from 15 commercial full-scale plants. The tested digestates originated from different substrates, with and without the pre-AD thermal hydrolysis process (THP). Moreover, a novel combined physicochemical parameter (C/N•ash) characterizing different digestate blends was identified by multiplying the C/N ratio with ash content of the dried solids. Using samples from 22 full-scale wastewater, food waste and co-waste plants, a linear relationship was found between C/N•ash and predicted cake solids for digestates with and without pre-AD THP. Pre-AD THP improved predicted cake solids by increasing the amount of free water. However, solids characteristics like C/N ratio and ash content had a more profound influence on the predicted cake solids than pre-AD THP and type of dewatering device. Finally, C/N•ash was shown to have a linear relationship to cake solids and reported polymer dose from eight full-scale pre-AD THP plants. In conclusion, we identified the novel parameter C/N•ash which can be used to predict dewatered cake solids regardless of dewatering device and sludge origin.

1. Introduction

As the world's population is growing the demand for dedicated sewage and organic waste treatment increases. Wastewater treatment generates two main streams of organic residues: primary sludge (PS) from initial sewage sedimentation and waste activated sludge (WAS) from aerobic biological treatment of the sewage liquid phase. WAS and PS with relatively low content of inorganic material (e.g. sand) are suitable as substrates for anaerobic digestion (AD), a biological process converting organic matter to renewable energy in the form of biogas. Organic waste from households or industry can be treated by co-digestion with PS and WAS or digested separately. Regardless of substrate, the digested residue (digestate) typically contains 95–98% water. To reduce transportation costs, efficient separation of water from solids (dewatering) is crucial. Thus, polyelectrolytes (polymers) are added to the digestate to bind particles into larger aggregates resulting in increased water release rate (Kopp and Dichtl, 2001b). However, large variations in dewatered cake solids are reported in literature despite similar AD configurations (Barber, 2016) implying that digestate physicochemical properties could be important in explaining these variations. Digestate dewatering and disposal can represent 30–50% of a full-scale plants' annual operating cost (Mikkelsen and Keiding, 2002). Consequently, predicting the

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https://doi.org/10.1016/j.watres.2019.04.037

expected cake solids is important for the design and optimization of full-scale AD plants.

Predictive models on dewatering have been developed or investigated by several authors (Nellenschulte and Kayser, 1997; Kopp and Dichtl, 2001b; Klinksieg et al., 2007; Skinner et al., 2015; To et al., 2018). Small particles have been negatively correlated to dewatered cake solids (Nellenschulte and Kayser, 1997). However, this does not include the effect of pre-treatments such as the thermal hydrolysis process (THP) that reduces particle size and still improves dewaterability (Neyens et al., 2004). Several methods of quantifying dewaterability have been studied including the use of filtration models (Skinner et al., 2015), replication of the full-scale process in the laboratory (To et al., 2018), rheological analysis (Klinksieg et al., 2007) and thermogravimetric analysis (TGA) (Kopp and Dichtl, 2001b). While most studies correlate their predictions to a small number of full-scale results, Kopp and Dichtl (2001b) found a linear relationship between their predicted cake solids and results from 33 full-scale plants digesting sewage sludge. The model assumes that polymer product and dosage in addition to selected dewatering device will be optimized by respective vendors in full-scale to reach the predicted maximum cake solids by TGA. However, it has not been validated for pre-treated digestates or other substrates than sewage sludge. Summarized, the literature on predictive models lacks comparison to a wide range of full-scale data or does not include the effect of different substrates and pre-treatments. All models are limited in the way that no single physicochemical parameter has been found linking dewaterability to a range of digestion substrates like PS, WAS or other organic wastes.

WAS containing up to 80% extracellular polymeric substances (EPS) has strong water holding capacities (Neyens et al., 2004; Skinner et al., 2015; Christensen et al., 2015). WAS has been described as a viscous gel-like material linked by hydrogen bonds and electrostatic forces (Markis et al., 2014). In contrast, PS behaves like a colloidal suspension where particles are linked by the much weaker van der Waals forces (Markis et al., 2014). Thus, WAS has higher viscosity (Hong et al., 2018) and less free water than PS, leading to poor dewaterability (Kopp and Dichtl, 2001a; Neyens et al., 2004; Christensen et al., 2015). The PS to WAS ratio is thus an important factor in digestate dewaterability. Despite the negative effect of WAS and EPS on dewaterability, no standard method for measuring EPS has been developed (Christensen et al., 2015). Alternatives such as the volatile solids (VS) concentration have been suggested (Skinner et al., 2015), but are not valid when digesting co-wastes such as food waste (Higgins and Rajagopalan, 2017). Additionally, the VS content in PS and WAS is similar (Suarez-Iglesias et al., 2017) and can therefore not explain the differences between these two substrates. However, a decreasing trend in dewaterability of digestates when the VS content increased was observed by Kopp and Dichtl (2001b) and they suggested this could be due to different behavior of organic and inorganic particles. Nicholson et al. (2018) investigated the effect of the carbon to nitrogen (C/N) ratio on dewaterability and found that an increase in C/N ratio coincided with increased dewaterability. However, both Kopp and Dichtl (2001b) and Nicholson et al. (2018) suggested that used alone neither VS nor C/N ratio could accurately predict dewaterability. Identifying a parameter that can describe these differences will help predict dewatered cake solids for conventional AD plants. However, the digestion substrate mix is often fixed, and to improve dewaterability pre-treatment such as the THP is needed (Neyens and Baeyens, 2003).

The THP as AD pre-treatment (pre-AD THP) improves dewaterability, but dewatering efficiencies depend on digestate characteristics and dewatering device (Barber, 2016). Improved dewaterability has been linked to reduced viscosity (Higgins et al., 2017). This could be due to the solubilization of EPS, weakening the water holding capacities of the flocs in WAS (Neyens et al., 2004). However, the mechanisms explaining the effect of pre-AD THP on dewatering and linking it to digestion substrates are not well documented.

In conclusion, a universal physicochemical parameter is needed to describe digestate dewaterability and the effect of pre-AD THP. In this study, we have developed a standardized and universal predictive model for dewatered cake solids by using a diverse sample set from full-scale plants. Based on digestates from a range of wastewater, food-waste and co-waste plants, the objectives of this study were: 1) to validate TGA as a method to predict digestate cake solids after full-scale dewatering for several digestion substrates with and without pre-AD THP, 2) to identify a universal digestate physicochemical parameter that can be used to predict cake solids, and 3) to investigate the effect of pre-AD THP on predicted cake solids.

2. Materials and methods

2.1. Samples

Digestates were collected from a total of 22 plants in Europe and the USA, with and without pre-AD THP, to study the effect of physicochemical properties and pre-AD THP on dewatered cake solids (Table 1). Additionally, full-scale dewatered digestates were collected at the outlet of the dewatering device at 15 plants to compare cake solids predictions by TGA to full-scale dewatering results (Plants A-O). Plants P-V did not run a dewatering process or used additives complicating direct comparison to the other dewatered digestates. Thus, comparison of TGA predictions and full-scale results for these plants were not valid or possible.

Digestates collected from Plants A-H in the United Kingdom (UK) were studied in most detail to identify physicochemical parameters affecting predicted cake solids. These plants used mesophilic anaerobic digestion (MAD) of sewage sludge with various pre-treatment methods, allowing comparison of conventional and pre-AD THP digestates. Two configurations of THP before AD (pre-AD THP) were sampled: THP treating both PS and WAS and only the WAS. In these plants, all THPs were operated at 165 °C for 30 min while pasteurization involved pre-treatment of PS and WAS at 70 °C for 1 h.

Data from Plants I and J have earlier been published by our research group (Svensson et al., 2018).

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

2.2. Thermogravimetric analysis

Thermogravimetric analysis (TGA) was used to determine the free water and predict dewatered cake solids in accordance to Kopp and Dichtl (2001b) with minor modifications described by Svensson et al. (2018). In brief; 100 mg samples were dried at 35 °C in a Netzsch Simultaneous TG-DTA/DSC Apparatus STA 449 F1 Jupiter® with a constant nitrogen flow of 20 mL/min. The drying curve was analyzed and the linear region prior to the change in drying rate due to the transition between free and interstitial water was identified. Linear regression analysis of this region identified the line defining the free water evaporation immediately before the transition. The deviation between the drying curve and free water defining line was calculated to identify the point between free and interstitial water. The drying curves are enclosed in Supplementary Material A. Calibration was done with mono-disperse silica particles of diameters 1.86 μm, 4.08 μm and 7.75 μm (Cospheric LCC, USA). Repeatability was investigated using five replicates on the

Table 1
Technical details of the plants sampled.

Plant ID	Thermal treatment	Digestion process Raw material	Continent	Dewatering device
Plant A	Pre-AD THP	MAD, sewage sludge	Europe	Hydraulic filter press
Plant B	Pre-AD THP	MAD, sewage sludge	Europe	Hydraulic filter press
Plant C	Pre-AD THP	MAD, sewage sludge	Europe	Belt press
Plant D	Pre-AD THP	MAD, sewage sludge	Europe	Belt press
Plant E	Pre-AD THP (WAS-THP)	MAD, sewage sludge	Europe	Hydraulic filter press
Plant F	Pasteurization	MAD, sewage sludge	Europe	Centrifuge
Plant G	None	MAD, sewage sludge	Europe	Centrifuge
Plant H	None	MAD, sewage sludge	Europe	Centrifuge
Plant I*	Pasteurization	MAD, food waste	Europe	Centrifuge
Plant J*	None	MAD, sewage sludge	USA	Centrifuge
Plant K	None	TAD, sewage sludge	Europe	Centrifuge
Plant L	Pre-AD THP	MAD, sewage sludge	Europe	Hydraulic filter press
Plant M	Pre-AD THP	MAD, sewage sludge	Europe	Centrifuge
Plant N	Pre-AD THP	MAD, sewage sludge and food waste	Europe	Centrifuge
Plant O	None	MAD, sewage sludge	Europe	Belt press
Plant P	Pre-AD THP	MAD, food waste	Europe	N/A
Plant Q	Pasteurization	MAD, pulp and paper sludge and fish waste	Europe	N/A
Plant R	None	MAD, sewage sludge	Europe	N/A
Plant S	None	MAD, sewage sludge	Europe	N/A
Plant T	None	MAD, sewage sludge	Europe	N/A
Plant U	Pasteurization	MAD, food waste and manure	Europe	N/A
Plant V	Pre-AD THP	MAD, food waste	Europe	N/A

*Data from Svensson et al. (2018).
N/A = not available.

same sludge sample. Predicted cake solids was found to be 40.6 ± 0.7 %DS.

2.3. Low-field nuclear magnetic resonance

Low-field nuclear magnetic resonance (LFNMR) was used to determine bound water in digestates from Plants A-H. LFNMR allowed a non-invasive measurement of the bound water in the digestate and, as opposed to dilatometric measurements, does not require any sample alteration such as freezing.

A Bruker mq20 minispec with a 0.47 T permanent magnet (Bruker, Billerica, MA, USA) was used to perform the LFNMR measurements previously described by Beck et al. (2018) to define bound water for Plants A-H. In brief; five mL of each sample was pipetted into a pre-weighed glass LFNMR tube and the total weight was recorded. The probe region of the LFNMR was stabilized at room temperature (22 °C) using a BVT 3000 nitrogen temperature control unit (Bruker, Billerica, MA, USA). Two minutes were allowed for the sample to equilibrate in the instrument before data acquisition. The Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence was used to measure the spin-spin relaxation time (T_2 relaxation time) of the samples with 32,000 echoes, gain 66 dB, 8 scans and a recycle delay of 5 s. The pulse separation (τ) for the measurements was optimized for the different sets of samples to allow full T_2 relaxation while minimizing τ. The CPMG decay curves were analyzed by continuous non-negative least squares (NNLS) fitting (Lawson and Hanson, 1974; Whittall et al., 1991) using PROSPA 3.2 (Magritek, Aachen, Germany). The NNLS fitting in PROSPA provides a continuous distribution of T_2 values. 512 data points were determined for each fit and a smoothing parameter of 0.5 was selected. For each peak, both T_2 values corresponding to maximum peak intensity and peak area were determined.

2.4. Moisture distribution

In this study the classification from Kopp and Dichtl (2001a) and Vesilind (1994) with three main water fractions was used; free water not bound by particles, interstitial water bound by capillary forces, surface water bound by adhesive forces and intracellular water including the water of hydration. The sum of surface and intracellular water was termed bound water. The amount of free, interstitial and bound water was determined by combining data from LFNMR and TGA analyses.

The free water was determined by TGA as described in section 2.2, and total water was determined by drying a sample at 105 °C until constant weight. Bound water was determined by LFNMR, where the area of the peak with shortest relaxation time was calculated in relation to the total area of all peaks. Interstitial water was calculated by subtracting the free and bound water from the total amount of water. Different pre-treatments and operational strategies of the AD process in Plants A-H lead to different digestate dry solids (DS) concentrations. To calculate and compare the moisture distribution between digestates all results were normalized theoretically to 3% DS to evaluate the percentage of free, interstitial and bound water. Normalization also allowed the comparison to previous results on moisture distribution applying TGA and dilatometric measurements (Kopp and Dichtl, 2001a).

2.5. Composition analysis

Digestates were analyzed for DS, VS, ash, carbon, nitrogen, iron and aluminum (Plants A-H) and acid detergent fiber (ADF) (Plants I, P and V) in the search for a physiochemical factor reflecting the digestion substrate that could predict cake solids after dewatering.

The DS, VS and ash concentration were measured gravimetrically in triplicates by drying a sample at 105 °C to constant weight followed by combustion at 550 °C.

Carbon and nitrogen were measured by combusting a dried sample (105 °C overnight) at 1150 °C with a constant flow of oxygen gas in a Vario El Cube elemental analyzer (Elementar Analysensysteme GmbH, Hanau, Germany).

PS/WAS ratios on dry solids basis were communicated by plant owner.

Iron and aluminum were analyzed by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) and Inductively Coupled Plasma – Optical emission Spectroscopy (ICP-OS). Samples were acidified and digested at 85 °C overnight before being analyzed by ICP-MS or ICP-OS.

ADF was analyzed according to manufacturer's recommendations using an Ankom[200] Fiber Analyzer (ANKOM Technology, Macedon, New York, USA) with F58 filter bags.

2.6. Statistical analysis

Principal Components Analysis (PCA) was performed on physicochemical parameters from Plants A-H (Supplementary Material B, Table SB1) using the software Past (Hammer et al., 2001). The correlation matrix was used since the variables were measured in different units.

3. Results and discussion

3.1. Predicted cake solids by TGA compared to full-scale results

Full-scale digestates and dewatered digestates originating from several different substrates, with and without pre-AD THP were used to validate the TGA as a good method for prediction of cake solids.

The cake solids from 15 full-scale dewatered digestates were successfully predicted by TGA and ranged from 17 to 34% DS ($R^2 = 0.90$, Fig. 1A), which is in line with previous findings from centrifuge dewatering by Kopp and Dichtl (2001b) ($R^2 = 0.92$), despite three different dewatering devices being used in the full-scale plants (centrifuge, belt press and hydraulic filter press). Some authors challenge the validity of drying tests, such as TGA, because the results depend on several factors such as material, sample size and drying conditions (Vaxelaire and Cezac, 2004). However, when calibrating with mono-disperse silica and using the same drying conditions and sample size, these issues were overcome for our samples. The samples included digestates from digesters treating pure food-waste, sewage sludge and a blend of sewage sludge and food-waste (Fig. 1A). Additionally, eight plants had pre-AD THP installed. Hence, we conclude that TGA is a reliable method for predicting dewatered cake solids on digestates originating from different substrates and with pre-AD THP.

Because dewatering devices function differently, one explanation for the difference in cake solids is the use of different dewatering devices. To investigate the dewatering devices' impact on final cake solids, we therefore grouped centrifuges, belt presses and hydraulic filter presses to determine if dewatering device would result in different correlations to the free water fraction measured by TGA (Fig. 1B). Grouping the results and applying linear regression analysis revealed that 7% higher cake solids were achieved with centrifuges compared to belt presses, although having similar free water content (Fig. 1B). This observation corresponds well with literature where centrifuges were reported to achieve 2–7% higher cake solids compared to belt presses (Novak, 2006). Samples from hydraulic filter presses were 9% dryer than those from centrifuges, although having similar free water content. According to Kopp and Dichtl (2001b) all the free water measured by TGA are removed in full-scale centrifuges. Hence, this implies that some of the interstitial water is accessed with a hydraulic filter press. Other authors have argued that some interstitial water can also be removed in dewatering of sludge by centrifugation or filtration (Vesilind, 1994; Novak, 2006). However, published full-scale data on hydraulic filter presses compared to centrifuges and belt presses in sludge dewatering were not found. Nevertheless, industrial testing supports that hydraulic filter presses access more water than centrifuges and belt presses (Thunberg, 2010).

The largest influence of dewatering device in the cake solids range tested would be 5% DS, comparing a belt press and a hydraulic filter press. Although dewatering device influenced the achieved cake solids, the variation across the data set (17–34% DS) cannot be explained by dewatering device. Hence, the difference observed in cake solids (17% DS) must be determined by digester substrate or operation. In the following section we therefore focused on identifying a universal physicochemical parameter, with the aim of relating this parameter to the water holding properties of digestates.

3.2. Digestate physicochemical properties

The current body of literature on sewage sludge dewatering suggests that the PS/WAS ratio, the amount and composition of the organic matter (VS, C/N), the inorganic matter and the moisture distribution are all important parameters influencing dewatering. This study aimed at identifying a single parameter that could describe digestate dewaterability. Eight plants (Plants A-H) were selected for detailed analysis of different physicochemical parameters. The feedstock of these plants spanned a wide range of PS/

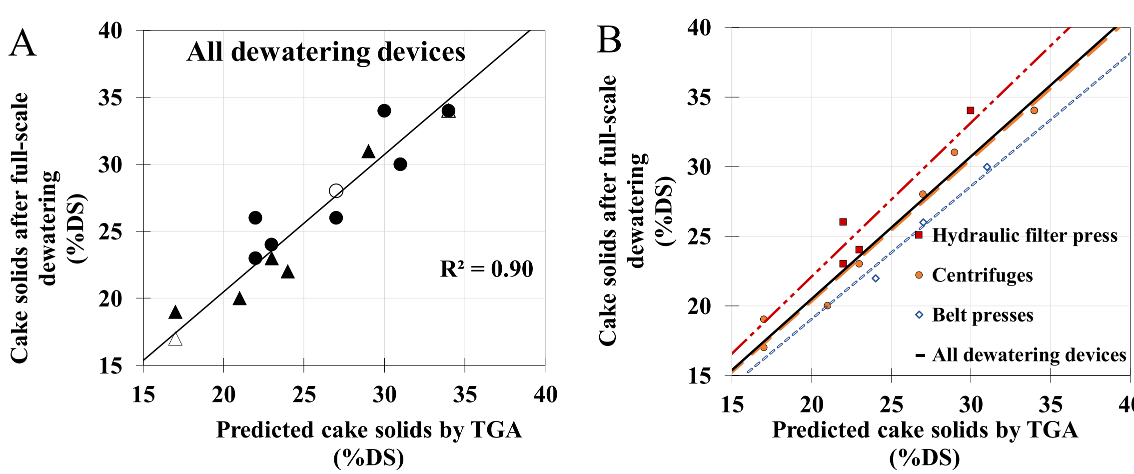

Fig. 1. Cake solids predictions by TGA compared to cake solids after full-scale dewatering for all dewatering devices tested (A). Data points in white triangles were earlier published by Svensson et al. (2018). Δ = conventional food waste digestion, ▲ = conventional digestion of sewage sludge, ● = pre-AD THP of sewage sludge, ○ = pre-AD THP and co-digestion of food waste and sewage sludge. Cake solids predictions by TGA and full-scale results from Fig. 1A were grouped into different dewatering devices with their respective linear regression lines fit to intercept at 0 (Fig. 1B): belt presses ($y = 0.95x$, $R^2 = 0.97$), centrifuges ($y = 1.02x$, $R^2 = 0.98$), hydraulic filter presses ($y = 1.11x$, $R^2 = 0.90$) and all dewatering devices ($y = 1.02$, $R^2 = 0.90$).

WAS ratios and different pre-treatment methods were applied.

The physicochemical characteristics of digestates from Plants A-H (Supplementary Material B, Table SB1) were combined in a principal component analysis (PCA) to identify the influence of various parameters on the free water content (Fig. 2). Two principal components (PC) described 87% of the variance in the dataset. For PC 2 (15%) the variance was mostly related to the moisture distribution (free, interstitial and bound water). Large amounts of free water correlated negatively with large amounts of interstitial and bound water similar to findings by Kopp and Dichtl (2001a). PC 1 described 72% of the variation and was related to physicochemical parameters typically measured at WWTPs or in commercial laboratories and the ratio of PS/WAS.

Plants A, B and E (Fig. 2, QA) and Plant H (Fig. 2, QC) had the highest amount of VS, carbon and nitrogen in the digestates. In addition, these plants had high amounts of WAS compared to PS. High amounts of VS and WAS have been shown to correlate negatively with dewatering performance (Kopp and Dichtl, 2001a, 2001b; Skinner et al., 2015). Although similar in VS, carbon and nitrogen content, Plants A, B and E groups in QA while Plant H is found in QC. The reason for this could be the application of THP in Plants A, B and E giving higher amounts of free water compared to Plant H.

High concentrations of iron and aluminum, ash, high PS/WAS ratio and high carbon/nitrogen (C/N) ratio were found for plants in QB and QD. These parameters had a positive impact on free water and hence the predicted cake solids (Fig. 2, PC1). Plant C, having the highest C/N ratio and concentration of iron and aluminum was also the plant with the highest amount of free water in the digestate (Fig. 2, QB).

The moisture distribution was mainly described by PC2 which explained only 15% of the variance in the data-set. Additionally, measuring moisture distribution requires instruments not normally present at WWTPs or in commercial laboratories. The physicochemical parameters primarily described by PC1 were therefore considered more relevant and useful.

The negative axis of PC1 is described by VS, carbon and nitrogen all representing the organic fraction of the sludge. The positive axis of PC1 is described by a combination of organic (C/N) and inorganic fractions (ash, iron and aluminum). Literature suggests that both organic and inorganic sludge content is important for the dewatering performance (Kopp and Dichtl, 2001b; Skinner et al., 2015; Miryahyaei et al., 2019) Thus, the parameters described by the positive PC1 axis are potential predictors of dewatering and therefore discussed in more detail below.

The role of cations in dewatering has been studied by several authors (Higgins and Novak, 1997; Park et al., 2006; Ngwenya et al., 2018). The divalent bridging theory (Higgins and Novak, 1997) linked the charge of divalent cations to the stabilization of sludge flocs and improved dewaterability by binding to the negatively charged EPS and particles. Ngwenya et al. (2018) also proposed the same mechanisms for monovalent cations. The ratio between monovalent and divalent cations has been shown to correlate with polymer dosage and dewaterability across multiple substrates (Higgins and Rajagopalan, 2017). The relatively high concentrations of iron and aluminum (Supplementary Material B, Table SB1) probably arise from the addition of inorganic coagulants in the wastewater treatment plant (WWTP). As pointed out by Park et al. (2006) inorganic cations might be better expressed as a fraction of the ash.

The positive impact of low volatile solids (VS) content and hence high ash content on predicted cake solids (Fig. 2) has also been reported by others (Skinner et al., 2015; Kopp and Dichtl, 2001b). Kopp and Dichtl (2001b) explained the positive influence of ash by the density difference between organic- and inorganic particles and the binding of more water by capillary forces to organic particles. Miryahyaei et al. (2019) showed that the addition of up to 0.07 g inert material/g WAS reduced viscosity of the digestate and improved dewaterability. However, addition of food waste low in ash content has also been shown to improve dewaterability of sewage sludge (Higgins and Rajagopalan, 2017). Although there seems to be an agreement in literature that the ash content plays a role in dewatering, an additional factor is needed to correct for the differences seen between PS and WAS and when adding a co-

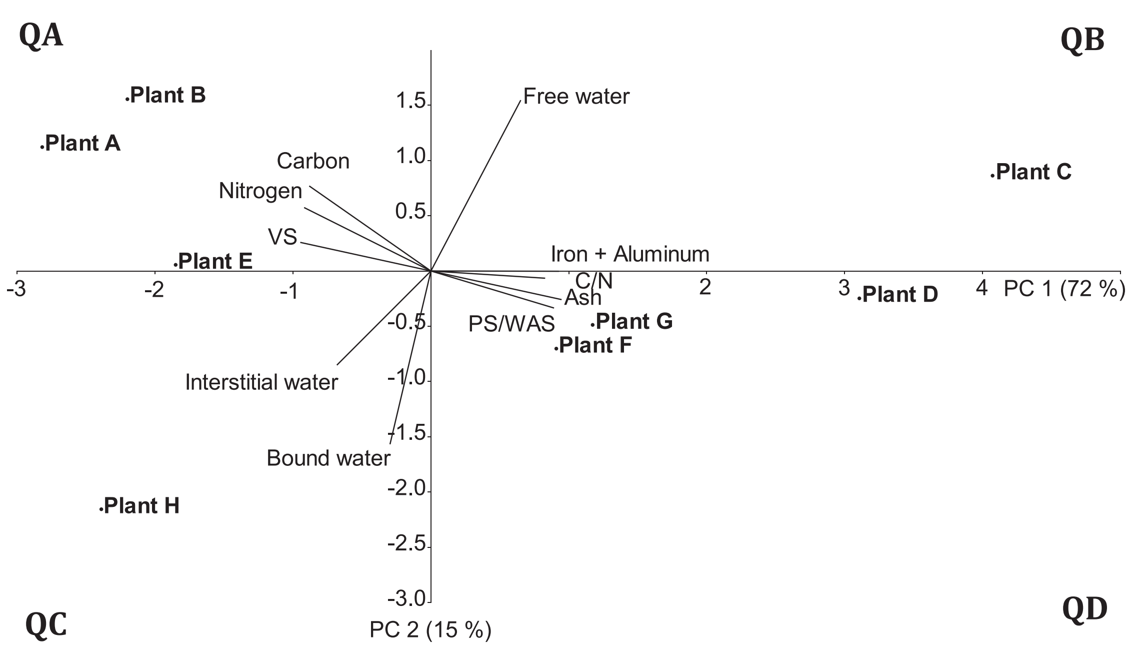

Fig. 2. PCA of physicochemical parameters and moisture distribution for Plants A-D (THP), Plant E (WAS-THP) and Plants F-H (None-THP).

substrate. This factor could be the C/N ratio. Several authors have observed that WAS contains more nitrogen and protein than PS and that WAS dewaters less than PS (Kopp and Dichtl, 2001a; Suarez-Iglesias et al., 2017; Higgins and Rajagopalan, 2017). The C/N ratio of PS was found to be substantially higher than WAS (Higgins and Rajagopalan, 2017; Nicholson et al., 2018) and was suggested to be an indicator of EPS (Nicholson et al., 2018). In addition to the difference observed in C/N of PS and WAS, Svensson et al. (2018) found a clear difference between C/N ratios of source separated food waste (SSFW) digestate and WAS digestate. The SSFW digestate dewatered to 34% DS, outperforming the WAS digestate which dewatered to 17% DS. Hence, the C/N ratio reflects the superior dewatering performance of PS and food waste compared to WAS. The amount of PS and WAS going to digestion can be challenging to accurately predict in full-scale as this factor depends on flow meters and manual sampling conducted at sometimes irregular intervals. Therefore, we propose to use C/N as a substitute for the PS/WAS ratio as this is a more general factor that also can be used for other substrates such as food waste.

Summarized, we identified four factors influencing dewatering positively; C/N, PS/WAS, ash and iron and aluminium. However, since iron, aluminium and other cations are a part of the ash, and C/N can be used as substitute for the PS/WAS ratio, we suggest only using two factors when relating digestate physiochemical properties to the free water and predicted cake solids: C/N ratio and ash. These two factors have been studied separately in relation to dewatering, but alone any of these two parameters cannot accurately describe the variance in dewatering. In this study we therefore tried combining these parameters to investigate if the effect of both inorganic- and organic material could explain predicted cake solids. Compared to organic material such as EPS, ash has a low water holding capacity which makes it an important parameter to include when assessing dewaterability. Additionally, inorganic compounds have also been shown to act as a skeleton adding a more rigid and incompressible structure to the sludge rendering it easier to mechanically dewater (Qi et al., 2011). This effect, including improved drainage and passage of water, is probably more important for dewaterability than just the low water holding capacity of ash (Qi et al., 2011). The beneficial effect of cations beyond contributing with fixed solids was also observed by Alm et al. (2016) measuring less water per volatile solids in dewatered cake when adding $FeCl_3$. Therefore, to combine the effect of the organic and inorganic composition of the digestate we multiplied the two factors together to create one combined factor, denoted C/N•ash. Both factors point in the same direction, i.e. both high C/N and high ash indicate low water binding and thus good dewaterability. We then investigated its correlation with predicted cake solids.

3.3. Predicting cake solids from physicochemical parameters

The sample set (Plants A-V) was used to study the correlation between C/N•ash and predicted cake solids by TGA on a broad range of digestion substrates (Fig. 3). The test matrix included digestion substrates such as sewage sludge, pulp and paper sludge, fish waste, food waste and manure.

Generally, the data showed a linear relationship between C/N•ash and predicted cake solids for all plants ($R^2 = 0.65$). However, when separating the pre-AD THP plants and conventional AD plants a much stronger linear relationship was obtained ($R^2 = 0.91$ and $R^2 = 0.93$). Two equations were identified by linear regression to predict dewatered cake solids of conventional and pre-AD THP digestates by using C/N•ash (Fig. 3). The results show that the variation in cake solids can be described by digestate physicochemical properties defined by C/N•ash.

The difference in predicted cake solids between the food waste plants (Fig. 3) cannot be explained by different PS/WAS ratios. Svensson et al. (2018) found a higher concentration of acid detergent fibers (ADF) in SSFW digestate compared to WAS digestate. Increasing ADF concentrations in the food waste digestates (Plants I, P and V) corresponded with increasing cake solids and C/N ratios in this study (data not shown). Thus, the C/N•ash also reflects

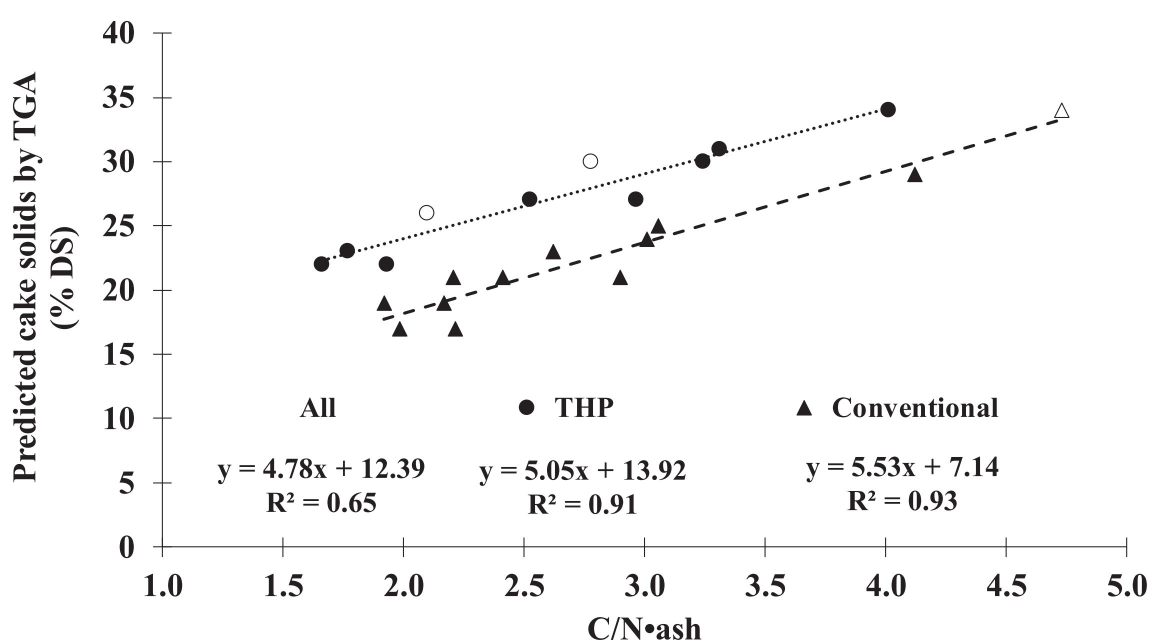

Fig. 3. The mass C/N ratio was multiplied with the relative content of ash (mass fraction), both measured on dry solids basis. The C/N•ash was compared to predicted cake solids by TGA for sewage sludge, food waste and co-waste AD digestates from conventional and pre-AD THP plants. Results from linear regression analysis of all, conventional and pre-AD THP plants are displayed. Pure food waste AD plants are indicated with open symbols. Data for Plant I (C/N•ash = 4.7) and J (C/N•ash = 2.2) have earlier been published by Svensson et al. (2018).

physicochemical properties in food waste digestates that are related to predicted cake solids.

C/N described the water binding capacity of the organic fraction. The amount of ash corrects for the inorganic material with low water binding capacity and skeleton building mechanisms. The combined C/N•ash was found to successfully describe the water binding capacity of digestates. The results suggest that the negative effect of high amounts of WAS can be counteracted if high amounts of ash is present in the digestate.

Overall, these results provide a novel correlation that can be used to predict the dewatered cake solids of conventional and pre-AD THP digestate from the physicochemical parameter C/N•ash. The two equations (Fig. 3) also make it possible to study how the installation of a pre-AD THP will influence the predicted cake solids.

3.4. Effect of pre-AD THP on predicted cake solids

The linear regression analysis of conventional and pre-AD THP plants (Fig. 3) was further used to study the effect of pre-AD THP on predicted cake solids.

The pre-AD THP changes the moisture distribution and increases the amount of free water for a given sludge blend, thereby increasing the dewatered cake solids (Fig. 3). In the C/N•ash range studied (1.7–4.7) the pre-AD THP improved dewaterability by 5–7% DS depending on the digestate. However, the difference in cake solids described by C/N•ash was 17% DS. Hence, digestate physicochemical properties have a bigger effect on the predicted cake solids than the application of pre-AD THP.

3.4.1. Relating physicochemical parameters to full-scale dewatered cake solids and polymer dose

The main goal of the dewatering process is to reduce the wet mass of cake by achieving the highest possible cake solids concentration. On the other hand, the amount of polymer used to achieve the desired cake solids is also of economic importance. The polymer dose (kg active substance (AS)/ton DS) and measured cake solids for eight full-scale pre-AD THP dewatering processes (Plants A-E and L-N) were compared to C/N•ash to relate C/N•ash to full-scale dewaterability (Fig. 4).

Linear relationships were found for both cake solids ($R^2 = 0.79$) and polymer dose ($R^2 = 0.90$) as a function of C/N•ash (Fig. 4). The highest reported polymer dose was at pre-AD THP plants digesting more WAS than PS (C/N•ash ≈ 1.7–1.8), and the lowest reported polymer dose from a plant digesting only PS (C/N•ash ≈ 4) (Fig. 4). This is in agreement with literature where WAS was readily solubilized in the THP compared to PS (Suarez-Iglesias et al., 2017), and increasing concentrations of soluble biopolymers have been linked to increased polymer dosage in sludge dewatering (To et al., 2018). The results show that, in addition to being correlated to the free water fraction measured by TGA, the C/N•ash is highly relevant to predict the full-scale dewatering process, including both cake solids and polymer dose for pre-AD THP plants.

3.5. Practical applications

Although the importance of the PS/WAS ratio is known, in full-scale operation the calculation of this ratio can be challenging due to dependence on flow meters as well as manual sampling by plant operators. Change in plant operation due to seasonal weather conditions, variation in co-waste quality and the digestion process itself also make it difficult to predict the effect of the substrate on digestate dewatering. More controlled conditions can be applied in laboratory studies, but comparison to full-scale and other laboratory studies can be difficult due to the lack of standardized analytical methods (To et al., 2016). In addition, large variations in sludge characteristics can also make it challenging to compare sludge blends used in different studies (To et al., 2016). For instance, it is known that sludge age of WAS will change the dewatering performance (Barber, 2014), a variable that is not adequately described when using the PS/WAS ratio. This study has identified a physicochemical parameter (C/N•ash) that overcomes these challenges, and will make it easier to compare the sludge blends used in different dewatering studies.

In the industry the C/N•ash will give realistic expectations for the dewatering process. This can support cost/benefit analysis prior to investments in several ways. As an example, for a pre-AD THP plant, the dewatering process can result in:

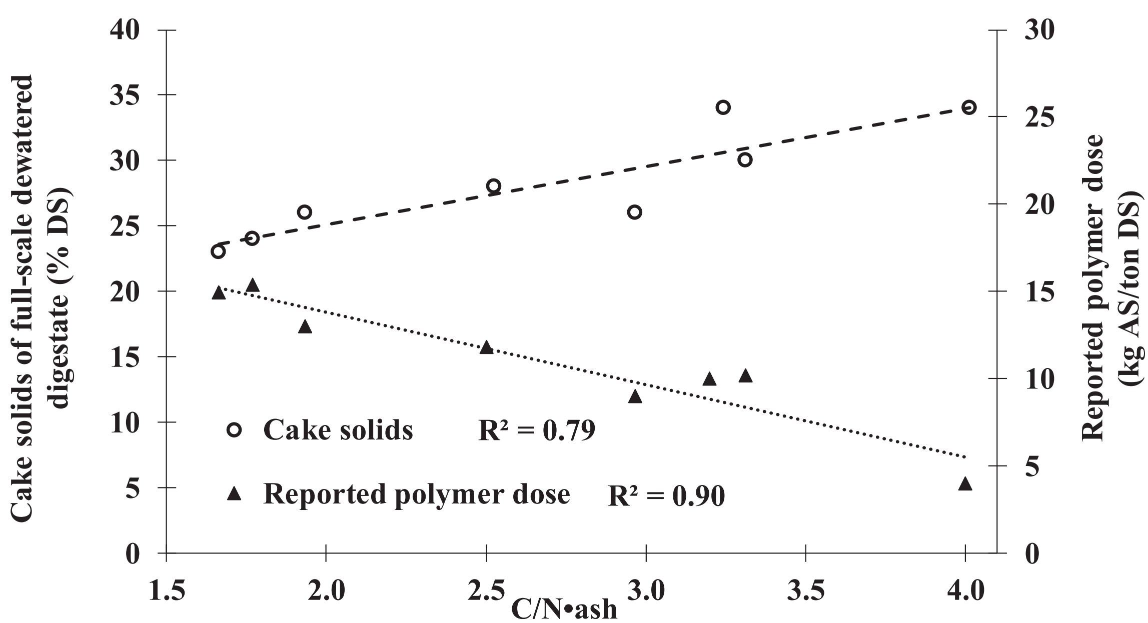

Fig. 4. The relationship between reported polymer dose (kg Active Substance (AS)/ton DS), full-scale dewatered cake solids (%DS) and digestate physicochemical parameters described by C/N•ash for eight full-scale pre-AD THP plants.

1) 23% DS in cake solids using 16 kg AS polymer/ton DS (Fig. 4, Plant A, C/N•ash = 1.7), or
2) 34 %DS in cake solids using 4 kg AS polymer/ton DS (Fig. 4, Plant M, C/N•ash = 4).

The different digestates described by C/N•ash will thus predict very different operational budgets for a plant, including both expenses for polymer and disposal (Boráň et al., 2010). Consequently, including C/N•ash in economic analysis can identify if optimization of the wastewater treatment process is necessary to maximize the amount of PS in the AD substrate. The C/N•ash correlation is thus a powerful tool in the planning and budgeting of full-scale plants (Piao et al., 2016).

In greenfield projects testing, demonstration and comparison of relevant dewatering devices (Guilayn et al., 2019) are done on existing plants. C/N•ash can therefore help identify suitable digestates for testing. It can also eliminate the need for full-scale testing completely, saving valuable time and money in an investment process.

Process guarantees, including dewatering expectations, are commonly set years before they must be demonstrated in the actual set-up. If discrepancies are observed between expected and actual values after project completion, C/N•ash can be used to identify if the dewatering process is not performing according to expectations or if the sludge blend has changed.

4. Conclusions

This study presents novel insight regarding the impact of digestate physicochemical properties and pre-AD THP on dewatered cake solids. The following conclusions were made:

1. TGA is a good method to predict full-scale dewatered cake solids. The method was valid for digestates from sewage sludge, food waste and co-digestion of these substrates, as well as pre-AD THP digestates.
2. A universal factor describing digestion substrates was found by multiplying the C/N ratio by the ash content of digestate dry solids (C/N•ash).
3. Strong linear relationships were found between C/N•ash and predicted cake solids by TGA for 22 full-scale plants when separating conventional and pre-AD THP digestates. Dewatered cake solids of both conventional and pre-AD THP digestates can thus be predicted from C/N•ash.
4. Pre-AD THP improved predicted cake solids of digestates by increasing the amount of free water.
5. C/N•ash showed linear relationships with both dewatered cake solids and polymer dose of full-scale pre-AD THP plants.

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

This work was financially supported by the Research Council of Norway (Grant no. 258749). Plants providing samples and support during the testing are greatly acknowledged for their efforts in this project. We thank Kine Svensson for comments and proof-reading the manuscript.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.watres.2019.04.037.

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