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Effect of digestate disintegration on anaerobic digestion of organic waste

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Abstract

Recently, digestate disintegration gained interest as an alternative strategy to feedstock pretreatment for anaerobic digestion. This study evaluated the effect of three different digestate disintegration methods (hydrogen peroxidation, ozone treatment and ultrasound) on manure digestate, potato waste digestate and mixed organic waste digestate. Lab-scale anaerobic digestion experiments were carried out by adding disintegrated digestate to the related substrate and inoculum with

simulated recycle ratios of 0.2 and 0.5. Ultrasound disintegration of potato waste digestate yields 22.5% increase in biogas production. An increase in biogas production was linked to the treated digestate amount and the treatment dosage. First order model was used to investigate the effect of digestate disintegration on the first order reaction rate constant (k). The decrease in k and increase in biogas production were linearly correlated. This correlation was explained by the increased bioavailability of the organic matter and possible negative effects of digestate disintegration on the microorganisms.

Key words: Ultrasound, ozonation, peroxidation, first-order kinetics, biogas

1. Introduction

Anaerobic digestion (AD) is a biological process that converts organic waste into biogas (55-70% CH_4) by degrading the organic material into nutrient rich digestate (Appels et al., 2011; Maynaud et al., 2017). Manure, fruit, vegetable and yard waste, and agricultural residues can be considered as organic lignocellulosic wastes (biomass) which can be valorized by anaerobic digestion (Mu et al., 2017; Nasir et al., 2012; Nordell et al., 2016). However, lignocellulosic biomass often requires pretreatment prior to AD to decrease the size and increase the surface area of the biomass. Thus the hydrolytic microorganisms can attach more easily to (hemi-)cellulosic components of biomass, which is necessary for an efficient hydrolysis (Azman et al., 2015; Budzianowski, 2016). Biomass pretreatment to increase the biochemical methane potential (BMP) of the feedstocks has been intensively investigated by means of lab-scale experiments (Carrere et al., 2016). In these studies, chemical (e.g. hydrogen

peroxide, ozone, acids, ionic liquids), biological (e.g. fungi), physical (e.g. milling) or as multiple or combined pretreatments were used to reach increased biogas yields (Agbor et al., 2011).

Digestate is a solid end product of AD. In general, the digestate still contains residual degradable organic material which can be further valorized. Digestates can be valorized as fertilizer, which is considered an ideal method for reuse of nitrogen, phosphorus and potassium (Zhang et al., 2018). However, digestates from animal manure are still considered animal waste and not a product, limiting its use (Sigurnjak et al., 2017). Chen et al. 2012 conducted a life cycle energy and environmental assessment on biogas and digestate utilization. The results suggest that digestate reuse is of equal importance to biogas utilization (heating, illumination and fuel) in the total energy production of the system (Chen et al., 2012). Digestate however also contain residual organic content which can be further valorized by AD. A mean organic degradation rate of $78 \pm 7 \%$ (in VS; w:w) for agricultural feedstocks was reported for continuous reactors (Ruile et al., 2015). An almost complete degradation of an manure can only be achieved with and HRT of 100 days (Ruile et al., 2015). However, the biodegradability of the digestate is relatively low and the residual biodegradability should be increased via some methods, similar to pretreatment, so called digestate disintegration. Pretreatments of substrate before AD were thoroughly investigated as early as 1983 (Lane, 1983) On the contrary, digestate disintegration is a relatively novel concept. The first mention of pretreatment of mechanically separated digested cattle slurry was made in 2011 (Menardo et al., 2011). Digestate disintegration can be used to increase the biodegradability of a digestate in a way that digestate can serve as a

substrate for a second digester or can be recycled as an additional feed to the existing digesters to increase the overall biogas production (Lindner et al., 2015).

Recently, chemical, thermal and physical treatments have been evaluated as possible digestate treatments by several authors. These studies reported increased soluble chemical oxygen demand (sCOD) and enhanced biogas production after the digestate treatments. Although the positive effect of disintegration techniques on solubilization and biogas production has been investigated, the kinetics of AD and the relationship between the changes in first order kinetics and biogas potential is usually overlooked (Boni et al., 2016; Garoma and Pappaterra, 2018; Lindner et al., 2015; Menardo et al., 2011; Ortega-Martinez et al., 2016; Sambusiti et al., 2015).

Digestate disintegration can be used to remove nutrients from the digestate. In most of the cases, digestate contains higher ammonia levels, compared to its substrates (Möller and Müller, 2012), which can be inhibitory for AD (Yenigün and Demirel, 2013). Thus, ammonia removal by digestate disintegration can be beneficial for the overall treatment. For example, ozone microbubbles were effective at removing ammonia in surface and ground water (Khuntia et al., 2013), whereas ultrasonication (US) was an effective ammonia removal tool in livestock waste management (Cho et al., 2014).

Hydrogen peroxide (H_2O_2) and ozone (O_3) are both oxidative pretreatments that increase the (hemi-) cellulose availability by removing the lignin (Patinvoh et al., 2017).

Hydrogen peroxide and ozone are two oxidants used in the pretreatment of waste activated sludge (Guan et al., 2018; Yasar and Tabinda, 2010). Hydrogen peroxide is dosed as a liquid, while ozone is a gas which bubbles through the treated medium.

During hydrogen peroxidation and ozonation, highly reactive hydroxyl radicals are released and they subsequently react with the organic and inorganic compounds of the feedstock (Khuntia et al., 2013). Hydroxyl radicals, formed by ozone, are also known to oxidize ammonia to nitrate in waste water (Khuntia et al., 2013). However, the formation of nitrate in digestate can reduce the biogas formation of the treated digestate (Ghyselbrecht et al., 2017). On the other hand, digestates contain high concentrations of carbonate ions (CO_3^{2-}) which react with hydroxyl radicals and produce carbonate radical ions during the oxidative pretreatments via the following reaction (R1) (Mehrvar et al., 2001):

Weeks and Rabani (1966) determined that the decay of these carbonate radicals can most probably be described by reactions R2 and R3 (Weeks and Rabani, 1966):

Or

R1 through R3 explains how carbonate ions act as hydroxyl radical scavengers. This scavenging effect may decrease the efficiency of oxidative treatments (Mehrvar et al., 2001). Hence, the effect of oxidation reactions on digestate must be evaluated to get a clear insight on digestate treatments.

Ultrasonic (US) disintegration is another method that can be suitable for digestate treatment. Ultrasonication of a liquid creates cavitation bubbles which collapse

violently when they reach a critical radius. Two phenomena occur upon collapse: i) the particulate matter is solubilized in the presence of high shear forces and, ii) hydroxyl radicals are produced (Pilli et al., 2011; Tyagi et al., 2014). Ultrasonication of the digestate can cause an increase in soluble fraction and methane production potential of the biomass. Boni et al. (2016) showed this effect with a full-scale anaerobic digester, treating a mixture of organic waste and activated sludge (Boni et al., 2016).

This study focuses on digestate disintegration as a tool for enhancing biogas production of dairy manure, potato waste and mixed organic waste. Three different pretreatment techniques were evaluated as a digestate disintegration technique: hydrogen peroxidation (chemical), ii) ozonation (chemical), and iii) ultrasonication (physical). Hydrogen peroxide and ozone are two oxidants used in the pretreatment of waste activated sludge. The two oxidative techniques were chosen based on their difference in dosing style (Guan et al., 2018). Ultrasound was selected based on its proven ability to disintegrate organic matter in digestates (Boni et al., 2016). The first part of our study investigates the effect of digestate disintegration, by means of degree of disintegration, and on the anaerobic batch assays at different digestate recycling ratios. In this scope, fresh substrate, untreated digestate (inoculum) and treated digestate were used in anaerobic batch assays and biogas production profiles were monitored. The second part investigates the effect of digestate disintegration on the biogas production rate, which was estimated by fitting the first order (FO) model to the experimental data.

2. Material and methods

2.1 Substrate and digestate composition

The experiments were carried out with three different substrates and their corresponding digestates, all originating from full-scale digesters, located in Flanders (Belgium). The first digestate was collected from a mesophilic pocket digester in Rumst. The pocket digester has 100 m³ volume, 17 days of sludge retention time (SRT), operates at 37°C and the digester is fed with dairy manure. The second digestate was taken from a digester in Moeskroen. The digester has a volume of 10200 m³ and 51 days of SRT. The digester processes potato waste from a potato processing plant. This digester was operated at mesophilic temperature at 37°C. However, during the experimental phase, the company decided to switch to thermophilic digestion at 51°C, and hence our experiments were conducted with both mesophilic and thermophilic digestate. The last digestate was taken from a digester in Merksplas. This digester processes a stream of mixed organic waste at 53 °C. The digester has 8000 m³ volume and 32 days of SRT. The manure, potato waste and mixed organic waste digesters were referred as “MAN”, “POT” and “OW”, respectively. All the digestates were taken from the reactors no more than 5 days before the start of the experiments and stored at room temperature. These digestates were selected based on their parameters as detailed in Table 1. The selection was determined by the organic content (VS w:w %) and the TAN content and a comparison was made between these digestates. OW has relatively high values on both VS (4.1) and TAN (5.2 g N/l), while mesophilic POT scored the lowest on both parameters with 2.3% VS and 0.9 g N/l TAN. MAN was selected based on its relatively high VS-content (4.8%) and a medium TAN content (2.1 g N/l).

2.2 Experimental setup of the disintegration techniques

In this study, three different disintegration techniques were selected for digestate treatment: hydrogen peroxide (H_2O_2), ozone (O_3) and ultrasound (US).

2.2.1 Hydrogen peroxidation

A 30% H_2O_2 solution (VWR, Belgium) was dosed to the three digestates in doses of 5, 10 and 30 g H_2O_2 /kg TS. The reaction was conducted in 500 mL beakers at room temperature ($21 \pm 3^\circ\text{C}$) and constant stirring by magnetic stirrer bar (100 rpm) for two hours to allow the foaming to disappear.

2.2.2 Ozonation

The ozonation experiments were performed under constant stirring by magnetic stirrer bar (300 rpm), at room temperature ($21 \pm 3^\circ\text{C}$), in a lab-scale glass reactor with an active volume of 1.7 L which is connected to an ozone generator (Anseros, Tübingen, Germany). The ozone generator was fed with pure oxygen gas (Air Liquide, Schelle, Belgium) with a flow rate of 75 l/h (Fig. 1). The ozone concentration of the gas stream, leaving the generator was 48.53 g O_3 /Nm³. The O_3 concentration in the off-gas (i.e. gas exiting the reactor) was monitored by an M465 O_3 monitor (Teledyne, San Diego, USA). Silicon oil (Buchi, Flawil, Switzerland) was added to the digestates with a concentration of 80 μl /kg to prevent foaming. The digestates were treated with 5, 10 and 30 g O_3 /kg TS.

[Fig.1]

2.2.3 Ultrasonication

The US experiments were carried out in a 1 L glass reactor with an active volume of 0.75 L. The reactor was kept below 20°C with a water jacket around the reactor to prevent thermal effects and its content was stirred with a magnetic stirring bar at 300 rpm. The US equipment consisted of a HD3200 US generator (Bandelin, Berlin), with a frequency of 20 kHz and a maximum power of 150 W. The Sonoplus-497 US horn (Bandelin, Berlin) with TT13 tip (Bandelin, Berlin) was placed at a depth of 3 cm in the glass reactor. The specific energy (SE) applied to the digestates were 3000, 9000 and 15000 kJ/kg TS and were calculated according to the Equation 1:

$$SE = \frac{P \cdot t}{V \cdot TS} \quad (\text{Equation 1})$$

Where P (W) is the US power, t (s) is the reaction time, V (L) the volume and TS (%) the total solid content of the digestate treated. The sonication density and intensity were 133 W/L and 113 W/cm², respectively.

2.2.4. Degree of Disintegration

The Degree of Disintegration (DD) was used to assess the efficiency of the disintegration techniques. The DD was calculated with the Equation 2. sCOD_{treatment} refers to the soluble COD after treatment, while sCOD₀ refers to the soluble COD of the original sample and tCOD the total COD of the digestate.

$$DD_{COD} = 100 * \frac{sCOD_{treatment} - sCOD_0}{tCOD - sCOD_0} [\%] \quad (\text{Equation 2})$$

2.3 Analytical methods

The substrates and digestates were characterized before treatment for pH, totals solids (TS) content, volatile solids (VS) content, and alkalinity. These parameters were measured according to the Standard Methods (APHA, 2005). All samples were centrifuged (15 minutes at 26200 g) and filtrated through 0.60 μm filter papers (Macherey-Nagel, Düren) to obtain the soluble fractions. Total and soluble chemical oxygen demand (tCOD and sCOD, respectively) were measured with Hach Lange test kits (LCK-014). sCOD and TAN were measured before after treatment. TAN and Total Kjeldahl nitrogen (KJN) were determined using the Kjeldahl-method of consecutive mineralization (only for KJN, Büchi Kjelflex K-439), distillation (Büchi Kjelflex K-360) and titration (Metrohm 848 Titrino plus) (Sambusiti et al., 2015). The substrate and digestate properties are shown in Table 1.

2.4 Set-up of the biogas production experiments

The effect of the different disintegration techniques on the biogas production was assessed using anaerobic batch assays. The biogas batch assay consisted of 1 L glass reactors with active volume of 0.8 L. The Food to Microorganisms ratios (F:M, on VS-basis) were 0.8, 0.5 and 0.25 for MAN, POT and OW, respectively. The F:M ratios were tested experimentally to prevent instant acidification. A part of the digestate was disintegrated with the described methods to simulate an anaerobic digester with a recycle (R_{sim}). Either 20% ($R_{\text{sim}} = 0.2$) or 50% ($R_{\text{sim}} = 0.5$) of the digestate was treated and used as a feed in batch assays. The relatively high recycle rates were chosen to magnify the effect of the digestate disintegration on the subsequent anaerobic digestion. The digestion temperature was kept at $37 \pm 1^\circ\text{C}$ for MAN and mesophilic POT.

For the thermophilic POT and OW the temperature was set at $51\pm1^{\circ}\text{C}$ and $53\pm1^{\circ}\text{C}$, respectively. The stable temperature was obtained by a water bath. The total biogas production volume was measured gravimetrically by the water displacement method, corrected for the moisture level (6.25%) and expressed in standard temperature and pressure (0°C and 1 atm). A negative control was set up with only inoculum (i.e. untreated digestate) to assess the biogas production of the inoculum. The biogas produced from this negative control was subtracted from the results of the other biogas production experiments. The results of these experiments were compared with a reference reactor (REF) with fresh substrate and without treated digestate.

2.5 Determination of kinetic parameters

The first order (FO) model was used to investigate the effect of the disintegration method on the biogas production rate (Equation 3) where B is the produced biogas (mL/g VS), t is the time (days), P is the maximum biogas production (mL/g VS) and k is the first order reaction rate (d^{-1}).

$$B = P * [1 - \exp(-k * t)] \text{ Equation 3}$$

Parameter fitting was done by minimizing the standard error of regression (S), as shown in Equation 4, where S is the standard error of regression, y is the data point, y' is the fitted model point and N is the total number of data points.

$$S = \sqrt{\frac{\sum(y-y')^2}{N}} \text{ Equation 4}$$

2.6 Statistical analysis

All tests were done in triplicate unless otherwise stated. Analysis of Variance (ANOVA) was used to determine if the treatment had a statistically significant impact on the analyzed parameters. Statistical significance was established at the $p < 0.01$ level. Results were considered marginally significant at $p < 0.05$. The Pearson's correlation coefficient R_p was used to determine the strength of a linear relationship between two variables. This coefficient ranges between -1, indicating a perfect negative linear correlation, and 1 indicating a positive linear correlation. An R_p of 0 denotes the absence of a linear relationship.

3. Results and discussion

3.1 Effect of digestate disintegration techniques on the degree of disintegration

MAN, POT and OW digestates were treated with hydrogen peroxide, ozone and ultrasound at different doses to investigate their capacity to disintegrate the residual solid matter in the digestate. The DD was determined after each treatment (Fig.2). Hydrogen peroxide treatment resulted in a maximum DD of $5 \pm 1\%$ at 10 g H_2O_2 /kg TS, $8 \pm 3\%$ at 5 g H_2O_2 /kg TS and $8 \pm 1\%$ at 10 g H_2O_2 /kg TS for MAN, mesophilic POT and OW respectively. The effect of hydrogen peroxide treatment on disintegration was not significant ($p = 0.052$, 0.224 and 0.037 for MAN, mesophilic POT and OW, respectively). Ozone treatment of the digestates also showed similar results. The maximum DDs were $2 \pm 1\%$ at 10 g O_3 /kg TS, $20 \pm 5\%$ at 10 g O_3 /kg TS and $4 \pm 4\%$ at 4 g O_3 /kg TS for MAN, thermophilic POT and OW, respectively. These treatments were also not significant ($p = 0.921$, 0.354 and 0.450 for MAN, thermophilic POT and OW, respectively). These results showed that hydroxyl radical based treatments were not

efficient on digestate disintegration. Ultrasound disintegration had the most promising results of DD. The highest DD's were $14 \pm 4\%$ at 9000 kJ/kg TS, 25% at 15000 kJ/kg TS and $24 \pm 2\%$ at 15000 kJ/kg TS for MAN, mesophilic POT and OW respectively.

Ultrasound significantly disintegrated the digestate ($p = 0.008$ and 0.006 for MAN and OW respectively). The high standard deviations were due to the viscous nature of the digestates. Nevertheless, these results clearly show that ultrasound more effectively disintegrates digestates compared to hydrogen peroxide and ozone. No significant TAN removal was observed for all the tested digestate and treatments. The maximum TAN removal for hydrogen peroxide treatment was 0.2, 0.2 and 0.5 g N/ L for MAN, mesophilic POT and OW, respectively. Ozone treatment yielded a non-significant increase in TAN of 0.1 g N/ L, 0.1 and 0.5 g N/ L for MAN, thermophilic POT and OW, respectively. US disintegration released TAN of maximum 0.1, 0.1 and 0.2 g N/ L for MAN, mesophilic POT and OW, respectively, however these were all non-significant as well.

Hydrogen peroxide and ozone treatment utilizes the oxidative nature of hydroxyl radicals. Digestates contain carbonate ions between 2.43 and 20.92 g CO_3^{2-} /L. These carbonate ions are known radical scavengers (Mehrvar et al., 2001). Therefore, the low disintegration effect and non-significant TAN removal effect can be explained by the presence of the carbonate ions.

[Fig. 2]

3.2 Effect of digestate disintegration techniques on the biogas production

The treated digestates were used for lab scale anaerobic digestion experiments with fresh substrate and inoculum. The biogas production of the inoculum (i.e. the negative control) was measured and subtracted from the other experimental results. The treated digestate was added in different recycle ratios of R_{sim} 's of 0.2 and 0.5.

MAN digestate treated with H_2O_2 , O_3 or US with R_{sim} of 0.2 caused a maximum increase in biogas production of 8.5%, 11.2% and 4.4%, when treated with 5 g H_2O_2 /kg TS, 5 g O_3 /kg TS and 15 000 kJ/kg TS, respectively, which were all statistically non-significant (Table 3). MAN digestate treated with H_2O_2 or US with R_{sim} of 0.5 resulted in a marginally significant increase in biogas production of 12.3%, and 8.6%, when treated with 30 g H_2O_2 /kg TS and 15 000 kJ/kg TS respectively. MAN digestate treated with 30 g O_3 /kg TS and a R_{sim} of 0.5 resulted in a 13.1% increase in biogas production, however also this was not statistically significant.

OW digestate treated with H_2O_2 did not result in an increase in biogas production. Even more, the biogas production was 6.8% lower when the OW digestate was treated with 30 g H_2O_2 /kg TS for R_{sim} of 0.5. The same was observed with ozone treatment, which caused a maximum biogas production increase of only 0.9% when treated with 5 g O_3 /kg TS for R_{sim} of 0.2, and a maximum decrease of 1.2% at the highest dose of 30 g O_3 /kg TS for R_{sim} of 0.5. US treatment resulted in a maximum biogas increase of 0.4% at 3000 kJ/kg TS for R_{sim} of 0.2 and a maximum decrease of 4.8% when treated with 15000 kJ/kg TS for R_{sim} of 0.5. These results were all statistically non-significant and the results proved that no digestate disintegration could increase the biogas production of the OW digestate.

Mesophilic POT digestate treated with 5 g H₂O₂/kg TS for R_{sim} of 0.2 resulted in a 4.9% biogas increase, while 10 g H₂O₂/kg TS for R_{sim} of 0.5 resulted in a 4.3% increase. Higher dosages of hydrogen peroxide did not result in higher biogas productions. Ozonation of the thermophilic POT digestate, with a dose of 10 g O₃/kg TS caused an increase in biogas production of 24.8% and 26.6% for an R_{sim} of 0.2 and 0.5, respectively. Despite the high increases in biogas production for ozonation experiment, these increases were not significant due to the high standard deviations between the experiments (Table 3). The high standard deviations of the experiment were related to an operational problem of the ozonation reactor with POT digestate. This digestate, unlike the other two digestates, dried out and stuck to the ozone aeration pebble, causing a locally high ozone concentration, compared to the rest of the medium. Therefore, hydrogen peroxide and ozone treatment are not suitable digestate disintegration techniques according to these results and the technical operational difficulties during the experiments.

[Fig. 3]

All US treatments on mesophilic POT digestate resulted in significant higher biogas productions ($p < 0.01$). The highest increases were 91.0 ml/g VS (+12.4%) and 166.0 ml/g VS (+22.5%) at 15000 kJ/kg TS for an R_{sim} of 0.2 and 0.5, respectively. The increase in biogas production caused by US was positively correlated with the applied SE (Table 2). Higher SE's on POT digestate resulted in higher biogas productions at both simulated recycle rates. When comparing the results for equal SE's between the simulated recycle rates, it is clear that a higher R_{sim} resulted in a higher biogas

production increase. The increased biogas production by ultrasonication of food waste was also confirmed by Elbeshbisy and Nakhla (2011). They reported a 26% increase in biogas production at 5000 kJ/kg TS (Elbeshbisy and Nakhla, 2011).

There are clear differences when comparing the effect of US on the biogas production between the three different digestates. These differences can be explained by the untreated digestate properties. The biogas productions of the negative controls (i.e. untreated digestates) used for the US experiments were 101.6 ml/g VS, 85.5 ml/g VS and 40.7 ml/g VS for MAN, POT and OW respectively. The low biogas production of the untreated OW digestate indicated that the remaining VS and soluble COD could be inert. Biogas production did not increase due to the possible increased soluble inert COD. Especially, manure contains inert lignin and humic acid fractions. US treatment can increase the concentrations of humic acids in the liquid phase of the digestate (Lu et al., 2018), which inhibit the hydrolysis and methanogenesis step of the anaerobic digestion (Fernandes et al., 2015; Khadem et al., 2017). The TS content of a given medium is another important parameter to consider when interpreting the results of US disintegration. The TS contents for MAN-, mesophilic POT- and OW digestates were $6.4 \pm 0.1\%$, $3.4 \pm 0.1\%$ and $8.4 \pm 0.1\%$ respectively. A TS content between 2.3% and 3% was found to be the optimal range for waste activated sludge (WAS) US-disintegration. If the TS content is higher than this optimal range, the solids will absorb the acoustic energy and decrease the efficiency of the process (Tyagi et al., 2014). The POT digestate, however distinct from WAS, most closely resembles this optimal TS-content.

The US disintegration experiment results were linearly correlated with the biogas production experiments. The soluble COD of the POT digestate was measured once after applying 3000, 9000 and 15000 kJ/kg TS, resulting in an sCOD of 9.9, 12.4 and 14.1 g/l, respectively. A R^2 of 0.97 and 0.89 for R_{sim} of 0.2 and 0.5, respectively were obtained whilst plotting the sCOD results with the biogas production results after ultrasonication (Fig. 4). A similar correlation was also observed for US disintegration of anaerobic digestate by Boni et al. (2016). The authors correlated the initial soluble organic matter before AD to the finale methane production of ultrasonicated digestates from a full scale AD plant (Boni et al., 2016).

The slope of the R_{sim} 0.5 ($y = 23x + 602$) was higher than the slope of R_{sim} 0.2 ($y = 13x + 652$). The relatively low slopes of these equations support that a high increase in sCOD is required for a moderate increase in total biogas production. Since the increase in sCOD is usually achieved by applying a very high specific ultrasound energy, an excessively high energy input is needed for a significant biogas production increase. Under the experimental conditions outlined in this paper, and based on the results discussed, an sCOD above 16 g/l would only be obtained by using an excessive SE over 15 000 kJ/kg TS which would damage the US device and increase the operational costs. Thus, sCOD of 16 g/l was chosen as an upper limit of the system.

Predicting the biogas production using the above fitted equation, and with a R_{sim} of 0.5 yields 972 mL/g VS in total or a 236 mL/g VS increase compared to the reference, (+32%). This result is confirmed by the study of Boni et al. (2016) in which they calculated a maximum 30% biogas increase with US treatment of solid waste digestate.

[Fig. 4]

3.3 Kinetic parameters

The first order (FO) reaction model was used to estimate the FO reaction rate constant (k) and the biogas production (P). The k values can be used to define the effect of the digestate disintegration methods, the respective dose and the simulated recycle rate.

The cumulative biogas production of OW showed a steep initial rise in biogas production (Fig. 3). This was due to the low pH of 3.5 of the fresh OW substrate used for the biogas production experiments. The low pH indicates a high concentration of organic acids which are readily available for biogas production. This steep initial rise in biogas production is not in accordance to the FO model assumptions and, hence, the FO model did not converge on the OW biogas production. Therefore, OW results were omitted from this discussion. Table 4 presents the fitted parameters for MAN and POT. The first order reaction rate of all treated digestates decreased compared to their untreated references. For seven treatments, the decrease in reaction rates were statistically significant (Table 5). For US treated MAN digestate, the highest decrease in k (46%) was observed for an R_{sim} of 0.5 and treatment with 9000 kJ/kg TS. For the H_2O_2 and O_3 treatment for R_{sim} of 0.5, the k values decreased by 32.6% and 39.9% when MAN digestate was treated with 30 g H_2O_2 /kg TS and 30 g O_3 /kg TS, respectively. The decrease in k values was less for R_{sim} of 0.2, compared to R_{sim} 0.5. For an R_{sim} of 0.2, k values decreased by 8.5% and 16.7% when treated with 30 g H_2O_2 /kg TS and 30g O_3 /kg TS respectively.

For the POT digestate, the maximum decrease in k was found when treating the POT digestate with 15 000 kJ/kg TS. This decrease was 11.5% and 50.8% at R_{sim} or 0.2 and 0.5, respectively. At a constant R_{sim} , k decreased at higher treatment dosages. At the same dosages, the decrease in k was greater at high simulated recycle ratio's (Table 6).

The decrease in reaction rates was related to the disintegration effect of the treatments. Oxidative pretreatments (e.g. ozone treatment) introduce highly reactive oxygen species in the anaerobic environment, increasing the possible oxidation-reduction potential (ORP) which negatively affects anaerobic digestion and thus lowering the first order reaction constant (Amani et al., 2010).

The effect of US treatment on biogas production was shown by many authors. For example, Kim and Lee (2012) observed a seven-fold increase in methane productivity with 30% disintegration of wastewater sludge. A 50% disintegration resulted in a relatively lower methane production rates and lower methane yields (Kim and Lee, 2012). In the US treatment of waste activated sludge (WAS), sludge flocs can be disintegrated (Tyagi et al., 2014). On the other hand, some studies have shown that brief US exposures can negatively affect the microorganisms by disrupting microbial cell walls (Pilli et al., 2011). Hence, digestate treatments negatively affect the microorganisms present in the digestate in addition to releasing biodegradable organic materials from complex structures (e.g. cellulose from lignocellulose). These two effects are proportional to each other, depending on the digestate disintegration dosage. Indeed, a negative correlation ($k = b_0.P + b_1$) was observed between the increase in biogas production (P) and the decrease in reaction rate constants, k (Fig. 5).

With exception of ozonated thermophilic POT digestate ($R_{sim} = 0.5$), all the b_0 coefficients and Pearson's correlation coefficients were negative with eight out of the twelve cases yielding significant ($p < 0.01$) results (Table 6). The slope, b_0 , is steeper at R_{sim} of 0.5 compared to 0.2. A higher R_{sim} can increase the amount of solubilized material and potentially negatively affected microorganisms in the reactor by adding a greater amount of disintegrated digestate to the reactor. The highest coefficient of determination (R^2) was 0.96 when 50% of the POT digestate was treated with the US treatment. The highest, statistically significant value of b_0 was (-16×10^{-4}) for the ozone treated MAN, indicating that a small increase in P corresponds with a high decrease in k . Ortega Martinez et al. (2016) also observed an increase in biogas production and a decrease in FO reaction rate constants when steam explosion was used to treat digestate originating from a waste water treatment plant. The formation of more complex and recalcitrant compounds was hypothesized to be the cause of the decreased rate constants (Ortega-Martinez et al., 2016). However, no linear correlation between P and k was reported. Under the experimental conditions of Boni et al. 2016, the kinetic parameter R_m (the maximum CH_4 production rate) and, the lag phase (λ) varied over a relatively narrow range (i.e. 18.2 L CH_4 /kg VS.d and 5.9-6.4 days respectively). However, no correlation between the lag phase and the applied US energy was observed (Boni et al., 2016). The modified Gompertz equation used by Boni et al. presupposes a lag phase. No lag-phase was observed under the given reaction conditions in this paper.

[Fig. 5]

In this study, only ultrasound disintegration of POT-digestate yielded an increase of biogas production, with a maximum of 22.5% compared to the reference. However, an economic feasibility study of this systems is out of scope for this study. An investigation regarding the economic advantage of disadvantage of digestate disintegration should include a full cost-benefit calculation on the investment including the capital expenditures (CAPEX) and operating expenses (OPEX), as well as environmental and societal impacts. Furthermore, batch experiments do not provide the necessary experimental data for such a calculation.

4. Conclusion

This study investigated the effect of three different digestate disintegration techniques on the disintegration and biodegradability of three different anaerobic digestates. The treated digestates were anaerobically digested with different simulated recycle ratios. Ultrasound disintegration is the most suitable digestate disintegration technique compared to ozone and hydrogen peroxidation in terms of disintegrating digestates that increases the biogas production. A linear correlation was observed between decreasing reaction rates and the increasing biogas production amounts for each treatment. The maximum biogas production increase was 22.5% for ultrasonicated potato waste digestate. E-supplementary data of this work can be found in online version of the paper.

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Figure Captions

Fig. 1. Schematic representation of ozonation set-up.

Fig. 2. Degree of disintegration (DD) for A: H_2O_2 , B: O_3 and C: US treatments for manure (MAN) digestate, mesophilic and thermophilic potato waste (POT) digestate and mixed organic waste (OW) digestate. Error bars denote the standard deviation between experiments. ⁱ: Duplicate. ⁺: Performed once, error bars here denote the standard deviation of the measurements.

Fig. 3. Cumulative biogas productions for A) H_2O_2 treated manure (MAN) digestate, B) O_3 treated mixed organic waste (OW) digestate and C) ultrasound treated mesophilic potato waste (POT) digestate. The most distinct results are shown. The reactors are identified as follows: After the type of substrate, the first letter (H, O or U) indicates the treatment: hydrogen peroxide (H), ozone (O) or ultrasound (US). Directly after the treatment-letter, the respective treatment-dosage is present. The last number, after the dash, indicates the simulated recycle rate: 0.2 or 0.5.

Fig. 4. sCOD of ultrasound (US) treated mesophilic potato waste (POT) digestate as a function of biogas production $R(sim)$ indicates the simulated recycle rate. Error bars for sCOD represents the standard deviation of the COD measurements.

Fig. 5. First order reaction rate as a function of modelled biogas production for A) manure (MAN) digestate and B) potato waste (POT) digestate. Only correlations with significant decreases in k and significant Pearson's correlation coefficients are shown.

Table Captions:

Table 1: Substrate and digestate properties Sub and Dig refer to substrate and digestate, respectively, for manure (MAN), potato waste (POT) and mixed organic waste (OW). Meso POT dig is the mesophilic potato waste digestate whereas Thermo POT Dig is the thermophilic potato waste digestate.

Table 2: Experimental results of the biogas production (ml biogas/g VS) after digestate treatment of manure (MAN), potato waste (POT) and mixed organic waste (OW).

Treatment doses a-b-c are 5-10-30 g H₂O₂/kg TS, 5-10-30 g O₃/kg TS and 3000-9000-15000 kJ/kg TS for hydrogen peroxidation (H₂O₂), ozonation (O₃) and ultrasonication (US) respectively. REF is the reference reactor with fresh substrate and untreated digestate.

Table 3: ANOVA results of biogas production per treatment and R_{sim}. Significant changes are presented in bold.

Table 4: Results of the first order parameter fitting. Treatment doses a-b-c are 5-10-30 g H₂O₂/kg TS, 5-10-30 g O₃/kg TS and 3000-9000-15000 kJ/kg TS for hydrogen peroxidation (H₂O₂), ozonation (O₃) and ultrasonication (US) respectively. The number (0.2 or 0.5) represents R_{sim}. The standard deviation notes the deviation of the fitted parameters. MAN: manure, POT: potato waste.

Table 5: Results of ANOVA test (p-values) for the difference in the first order reaction rate (k). MAN: manure, POT: potato waste. Significant changes are presented in bold.

Table 6: Linear regression coefficients for $k = b_1 \cdot P + b_0$ with respective p-values, Pearson coefficient and coefficient of determination (R²). MAN: manure, POT: potato

waste, OW: mixed organic waste. Significant correlations are presented in bold.

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	Unit of measure	MAN Sub		MAN Dig		POT Sub		POT meso Dig		POT thermo Dig		OW Sub		OW Dig	
TS	%	9.85	±0.19	6.43	±0.04	14	±0.12	3.35	±0.07	3.43	±0.01	19.38	±0.05	8.39	±0.03
VS	%	7.91	±0.2	4.86	±0.06	13.1	±0.12	2.33	±0.08	2.50	±0.01	12.85	±0.04	4.12	±0.08
tCOD	g/l	#####	±9.37	47.6	±0.98	209	±3.9	36.1	±3.48	37.50	±0.36	204.20	±16.07	56.5	±0.97
sCOD	g/l	#####	±5.49	17.6	±0.14	86	±1.7	6.6	±0.69	18.2	±0.28	167.8	±12.97	21.3	±0.51
Kjeldahl-N	g N/l	4.03	±0.56	3.42	±0.06	2.52	±0.17	2.86	±0.05	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
TAN	g N/l	1.98	±0.03	2.09	±0.02	0.39	±0.05	0.92	±0.08	0.62	±0.02	0.43	±0.01	5.18	±0.16
Alkalinity	g CO ₃ ²⁻ /l	n.d.	n.d.	8.06	±0.16	n.d.	n.d.	n.d.	n.d.	2.43	±0.06	n.d.	n.d.	20.9	±0.09
pH	-	6.9	±0.2	8.0	±0.1	3.5	±0.1	7.4	±0.1	7.3	±0.2	3.5	±0.1	8.3	±0.1

n.d.: not determined

		REF		a-20		a-50		b-20		b-50		c-20		c-50	
MAN	H ₂	427.	±19.3	463.		468.	±9.7	455.		460	±6.6	446.	±16.	479.	±15.
	O ₂	2	3 ⁱ	7	±0.22 ⁱ	1	5	4	±12.3 ⁱ	0	9	65	9	60	
	O ₃	356.	±43.5	396.		364.	±32.	305.		323.	±51.	389.	±116	403.	±38.
	US	5	2	3	±27.27	9	30	8	±83.7	8	27	4	.9	4	43
POT	H ₂	241.	±4.10	240.		245.	±5.9	249.		216.	±24.	252.	±5.4	262.	±3.9
	O ₂	9		1	±6.84	7	2	1	4	9	34	6	1	6	4
	O ₃	712.	±15.2	727.		742.	±3.6	747.		708.	±22.	745.	±14.	734.	±6.9
	US	3	2	6	±7.34	9	1	6	9	9	39	6	29	4	4
OW	H ₂	636.	±123.	730		733.	±28.	794.		806.	±73.	n.d.	n.d.	n.d.	n.d.
	O ₂	9	30	4 ⁱ	±112.2	6	73	9	92	6	92	n.d.	n.d.	n.d.	n.d.
	O ₃	736.	±12.0	779.		859.	±9.5	823.		906.	±23.	827.	±4.1	902.	±4.9
	US	3	0	8	±3.26 ⁱ	4	9	8	6	6	8	4	8 ⁱ	4	0
US	H ₂	918	±18.6	897.		872.	±40.	n.d.		n.d.		918.	±4.7	855	±23.
	O ₂	8	1	±46.17	6	01	n.d.	n.d.	n.d.	n.d.	2	4	855	79	
	O ₃	918.	±18.4	926.		917.	±5.6	n.d.		n.d.		917.	±63.	907	±74.
	US	5	9	5	±14.92	4	2	n.d.	n.d.	n.d.	1	7	861.	±59.	845.
		888.	±18.4	891.		872.	±33.	n.d.		n.d.		861.	±59.	845.	±15.
		5	0	8	±31.54	2	59	n.d.	n.d.	n.d.	4	18	8	05	

i: duplicates

n.d.: not determined

R_{sim}	MAN		POT		OW	
	0.2	0.5	0.2	0.5	0.2	0.5
H ₂ O ₂	0.198	0.015	0.054	0.177	0.633	0.087
O ₃	0.492	0.221	0.385	0.123	0.953	0.940
US	0.924	0.016	0.001	0.000	0.591	0.165

		REF				a-0.2				a-0.5				
		P		k		P		k		P		k		
MAN	H ₂ O ₂	408.1	±13.9	0.138	±0.006	441.8	±0.1	0.132	±0.004	447.9	±6.9	0.122	±0.002	433.4
	O ₃	256.6	±2.8	0.198	±0.006	254.5	±4.3	0.186	±0.002	259.3	±12.4	0.163	±0.014	270.6
	US	253.7	±2.8	0.196	±0.006	263.1	±5.3	0.175	±0.006	297.6	±9.6	0.127	±0.010	276.9
POT	H ₂ O ₂	728.9	±9.8	0.476	±0.002	746.5	±6.5	0.435	±0.009	771.9	±2.1	0.401	±0.011	768.7
	O ₃	643.2	±59.9	0.206	±0.047	741.5	±104.6	0.196	±0.034	761.0	±30.7	0.195	±0.009	811.4
	US	757.1	±8.3	0.431	±0.003	820.6	±8.7	0.400	±0.004	926.0	±8.6	0.267	±0.006	856.6

R_{sim}	MAN		POT	
	0.2	0.5	0.2	0.5
H ₂ O ₂	0.371	0.000	0.005	0.001
O ₃	0.008	0.000	0.838	0.374
US	0.526	0.002	0.024	0.000

	R_{sim}	b_1 (*10 ⁻⁴)	p -value of b_1	Pearson's coefficient	b_0	p -value of b_0	R^2
MAN H ₂ O ₂	0.2	-0.4	0.797	-0.101	0.15	0.059	0.01
	0.5	-5.6	0.009	-0.741	0.08	0.001	0.55
O ₃	0.2	-11.0	0.001	-0.850	0.47	0.000	0.72
	0.5	-16.0	0.001	-0.887	0.58	0.000	0.79
US	0.2	-7.0	0.000	-0.926	0.37	0.000	0.86
	0.5	-10.0	0.002	-0.795	0.44	0.000	0.63
POT H ₂ O ₂	0.2	-6.9	0.006	-0.741	0.96	0.000	0.55
	0.5	-10.0	0.070	-0.539	0.27	0.024	0.29
O ₃	0.2	-0.9	0.617	-0.210	0.26	0.075	0.04
	0.5	0.6	0.737	0.131	0.15	0.278	0.02
US	0.2	-3.9	0.005	-0.801	0.72	0.000	0.64
	0.5	-8.4	0.000	-0.981	1.06	0.000	0.96

Highlights (max 85 characters incl. space)

- Ultrasonication of potato waste digestate yields 22% increase in biogas production.
- Digestate disintegration increases the biogas production with increasing dosage.
- Digestate disintegration decreases the first order constant with increasing dosage.
- Higher recycle rates of treated digestate increase the biogas production.
- Higher recycle rates of treated digestate decreases the first order rate constant.