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Research article

Thermal thickening of nitrified liquid phase of digestate for production of concentrated complex fertiliser and high-quality technological water

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ABSTRACT

Large production of liquid phase of digestate (LPD) characterised by low dry matter content increases the costs of LPD storage and transportation. Additionally, significant nitrogen losses result from LPD handling. The operation of biogas plants also requires large amounts of technological water, a scarce resource. The method of LPD nitrification and subsequent thermal thickening as a promising alternative treatment able to overcome those problems was investigated. The properties of thermally thickened nitrified LPD and of the distillate derived from vacuum evaporation of nitrified LPD were examined. The results show that over 99.9% of nitrogen was accumulated in thickened nitrified LPD. Total nitrogen (TN) concentration in thickened LPD ranged from 12 to 18 g/L depending on the distillate/thickened LPD volume ratio applied. The thickened LPD was also richer in other nutrients, which indicates its possible use as a concentrated complex fertiliser. TN concentration and conductivity in the distillate did not exceed 6 mg/L and 0.03 mS/m respectively, suggesting that the distillate may be useful as alternative source of technological water. It was concluded that the system consisting of nitrification and subsequent thermal thickening of nitrified LPD yields final products exhibiting significantly superior properties compared to thermal thickening of raw LPD, without requiring any additional chemical agents. The quality of these products is comparable to those obtained through advanced membrane processes.

1. Introduction

Digestate is a by-product of the anaerobic digestion of biodegradable materials in biogas plants (BGPs) (Al Seadi et al., 2013; Nkoa, 2014). In order to improve its management, digestate can be separated into solid and liquid phases (Monfet et al., 2018). The liquid phase of digestate (LPD) contains a relatively high concentration of nitrogen that usually ranges from several hundreds of milligrams to several grams per litre, where the predominant nitrogen form is total ammonia nitrogen (TAN, or the sum of $N\text{–}NH_4^+$ and $N\text{–}NH_3$) (Botheju et al., 2010; Vaneeckhaute et al., 2017; Shi et al., 2018). Besides nitrogen, digestate and LPD are rich in other important nutrients for plants such as phosphorus and potassium (Koszel and Lorencowicz, 2015; Vaneeckhaute et al., 2017; Bolzonella et al., 2018; Shi et al., 2018). For these reasons, the most common utilisation of LPD is the direct application on agricultural land as a fertiliser. LPD can also be re-fed to the anaerobic digester to reach the required value of dry matter (DM) content in the material entering the reactor (Al Seadi et al., 2013). In some cases, LPD is handled as wastewater when its nutrients cannot be efficiently utilised (Monfet et al., 2018). The use of LPD as fertiliser is strictly limited by legislation. In EU countries, the Nitrate Directive (91/676/EEC) protects ground and surface water from nitrate pollution by limiting the amount of LPD that can be applied to the soil (European Commission, 1991). Therefore, LPD must be stored in lagoons of a given BGP on a relatively long-term basis until its use (Al Seadi et al., 2013; Plana and Noche, 2016). LPD also contains relatively large amounts of ballast water. The direct application of LPD near BGP installations is often not possible, increasing storage and transportation costs (Rehl and Müller, 2011; Khan and Nordberg, 2019). Moreover, emission of ammonia during LPD storage and application may be high (Whelan et al., 2010; Nkoa, 2014; Nicholson et al., 2017; Perazzolo et al., 2017; Svehla et al., 2020). Such losses of nitrogen can lead both to a reduction of LPD fertiliser quality and pollute the environment. The use of LPD as diluting water to optimise DM content for biogas production creates various problems. Accumulation of ammonia may result from the recirculation of LPD into the digesters, leading to inhibition of microbial activity during biogas production (Hu et al., 2014; Drosg et al., 2015). Finally, a relatively large fraction of inert dry matter is unnecessarily recirculated into the

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reactor, thereby limiting the biogas plant capacity (Lindmark et al., 2011; Khan and Nordberg, 2019).

The summary above highlights the need for LPD treatment methods enabling the enhancement of nutrients concentration and minimisation of nitrogen losses and other problems resulting from the recirculation of LPD into digesters. Thermal thickening by vacuum evaporation represents a promising alternative (Bonmatí et al., 2003; Chiumenti et al., 2013; Guercini et al., 2014; Fechter and Kraume, 2016; Vondra et al., 2016, 2018bib_Vondra_et_al_2016bib_Vondra_et_al_2018) that produces thickened LPD that can be used as a concentrated complex fertiliser. Moreover, the recovered distillate can be used as technological water for the dilution of substrate entering the anaerobic digester, thereby reducing the risk of inhibition and undesirable recirculation of inert particles. On the other hand, during thermal thickening of raw LPD a significant proportion of TAN may be transferred into the distillate (Botheju et al., 2010; Guercini et al., 2014), which is undesirable as it reduces the suitability of the distillate as technological water as well as the amount of nitrogen conserved in thickened LPD. For those reasons, some authors proposed the accumulation of TAN in distillate instead of thickened LPD (Simonič and Simonič, 2018). However, acidification of raw LPD by addition of mineral acid before thermal thickening has often been reported as a way to optimise nitrogen conservation in thickened LPD (Bonmatí et al., 2003; Panvini et al., 2009; Chiumenti et al., 2013). Unfortunately, this method of pH adjustment may result in significant operational costs (Al Seadi et al., 2013; Guercini et al., 2014), as well as in an elevated risk of soil acidification from fertilisation with acidified thickened LPD (Rorison, 1973; Rice and Herman, 2012). Therefore, developing an alternative method for the minimisation of nitrogen transfer into the distillate during thermal thickening of LPD constitutes a relevant challenge. In laboratory conditions, it was found that nitrification is a reasonable pre-treatment of LPD before thermal thickening (Botheju et al., 2010; Svehla et al., 2017) by turning TAN into oxidised forms of nitrogen (N–NO$_3^-$ or N–NO$_2^-$) which are significantly less volatile. As a consequence of the nitrification process performed in the environment of LPD, pH may decrease from slightly alkaline values to 5.5–6.5 or even lower (Botheju et al., 2010; Svehla et al., 2017). Thus, volatilisation of ammonia during thermal thickening may be minimised even if a fraction of TAN is not converted into oxidised forms. The presence of N–NO$_2^-$, a form of nitrogen that can have toxic effects on plants (Oke, 1966), can be minimised by the regular control of the nitrifying reactor (Svehla et al., 2017).

This study investigates the basic properties of products of nitrified LPD thermal thickening. Nitrified LPD was produced in a laboratory nitrification reactor. Distribution of nitrogen between thickened nitrified LPD and distillate is reported and the suitability of thickened nitrified LPD as a complex fertiliser is discussed. The use of distillate derived from the process for the production of technological water was also investigated. It was concluded that the proposed two-stage treatment of LPD by nitrification and subsequent thermal thickening of nitrified LPD is suitable for full scale applications.

2. Materials and methods

2.1. *Nitrified LPD*

LPD was obtained from an agricultural BGP operated under mesophilic conditions and fed with cattle manure and slurry (around 70% of the raw matter weight of substrate for biogas production), corn silage (20%) and grass silage (10%). Separation of digestate into solid and liquid phases was performed with a press separator at the BGP. Raw LPD was exposed to a nitrification process in a 5-Litre continuously stirred tank reactor (CSTR) equipped with sedimentation tank operated as in Svehla et al. (2017). Nitrification biomass was recirculated from the sedimentation tank back into the nitrification CSTR, where no excess biomass was intentionally withdrawn from the system with the aim to maximise nitrification biomass retention. The nitrifying biomass was cultivated in the form of a suspension (activated sludge) in this system. The sample of nitrified LPD was collected from the effluent of the sedimentation tank. Basic characteristics of the nitrified LPD are shown in Table 1.

2.2. *Thermal thickening by vacuum evaporation*

Vacuum evaporation of nitrified LPD was conducted using a BÜCHI rotavapor R-215 (BÜCHI, France). The heating water bath (BÜCHI heating bath B-491, France) was set to 95 °C to facilitate evaporation. 200 mL of nitrified LPD and 10–12 drops of Burst® 8900 defoamer (BASF, Germany) were poured into a round bottom flask (boiling flask) for connection to the rotor-vac apparatus. The boiling flask was rotated continuously at 40 rpm, at a temperature set in accordance with the boiling point of water at a given vacuum level (65 ± 5 °C at 350 ± 40 mbar pressure) by means of a BÜCHI vacuum controller V-850 and vacuum pump V-700 (BÜCHI, France). Three evaporation treatments (Evaporation series 1, Evaporation series 2 and Evaporation series 3) characterised by the evaporation of 50%, 60% and 67% of the original sample volume were performed. Distillate/thickened LPD volume ratios were 1.0, 1.5 and 2.0 at Evaporation series 1, Evaporation series 2 and Evaporation series 3 respectively. Each evaporation series was replicated twice.

2.3. *Analytical methods*

The value of pH, electrical conductivity (EC), concentration of total solids (TS), total nitrogen (TN), TAN, N–NO$_2^-$, N–NO$_3^-$ and chemical oxygen demand (COD) were estimated in the samples of nitrified LPD, thickened nitrified LPD and distillate. The samples of nitrified LPD and thickened nitrified LPD were centrifuged at 9500 rpm at g-forces of 12.007 for 12 min using a Rotina 420 centrifuge (Andreas Hettich GmbH & Co. KG, Germany) before the measurement of TN, TAN, N–NO$_2^-$, N–NO$_3^-$. Additionally, the concentrations of Ca, K, Mg, P, S, Cd, Pb, As, Cr, Cu, Mo, Ni, and Zn were measured in the samples of thickened nitrified LPD to determine the nutrient and risk element content in the stream. For Evaporation series 1, the concentration of volatile fatty acids (VFAs) was determined in nitrified LPD, thickened nitrified LPD and distillate.

The concentrations of TAN, N–NO$_2^-$, N–NO$_3^-$, and COD were measured in accordance with standard methods (APHA et al., 2017) using a HACH DR/4000 photometer (Hach Lange, Germany). TN was measured by HACH method number 10071 using the same photometer. TS concentration was measured gravimetrically in accordance with standard methods (APHA et al., 2017). The value of pH was measured using a WTW pH 340i pH meter connected with pH-probe WTW SenTiX 21 (WTW, Germany). EC was determined by a conductometer inoLab Cond 730 (WTW, Germany). Ca, K, Mg, P, S, As, Cd, Cr, Cu, Mo, Ni, Pb and Zn were determined as in Zakova et al. (2016) using inductively coupled plasma optical emission spectrometry (ICP-OES, Agilent 720, Agilent Technologies Inc., USA) equipped with a two-channel peristaltic pump, a Sturman-Masters spray chamber and a V-groove pneumatic nebuliser. In this manner, the so-called pseudo-total elements content reflecting

Table 1
Basic characteristics of nitrified LPD. TS: total solids concentration. COD: chemical oxygen demand. TN: total nitrogen concentration.

Parameter (units)	Measure
pH (–)	6.1 ± 0.05
TS (g/L)	48.5 ± 3.49
COD (mg/L)	3760 ± 91
N–NO$_3^-$ (mg/L)	5510 ± 151
N–NO$_2^-$ (mg/L)	0.7 ± 0.26
TAN (mg/L)	8.9 ± 2.77
TN (mg/L)	5840 ± 170

the amount of elements extractable with aqua regia were analysed. The pseudo-total content of Hg was measured according to Sipkova et al. (2016) using inductively coupled plasma mass spectrometry (ICP-MS, Agilent 7700x, Agilent Technologies Inc., USA), equipped with an autosampler ASX-500, a three-channel peristaltic pump and a MicroMist nebuliser. VFAs were measured using a Thermo Scientific TRACE 1300 Gas Chromatograph (GC) (Thermo Fisher Scientific, UK) equipped with an FID detector. Samples were separated by a DB-FFAP capillary column (30 m, 0.25 mm internal diameter, 0.25 μm film thickness, Agilent Technologies Inc., USA) with hydrogen as a carrier gas. First, 800 μL of the sample were pipetted and mixed with 0.033 mL of H_3PO_4. Finally, 10 μL of the mixed sample were injected into the GC.

2.4. *Quantification of acid requirement for raw and nitrified LPD acidification*

A specific experiment to compare acid consumption during acidification of raw versus nitrified LPD at different required pH values was performed. Original pH values reached 8.3 and 6.1 for the samples of raw and nitrified LPD respectively. Samples were titrated with hydrochloric acid (0.1 mol/L) until the required pH value was achieved, and results were then recalculated.

2.5. *Calculations*

The percentage distribution of TN between the products of thermal thickening (thickened nitrified LPD and distillate) was calculated as:

$$\%\ TN\ (thickened\ LPD) = \frac{\rho_T(TN)\ .\ V_T}{\rho_{T(TN)}\ .\ V_T\ +\ \rho_D(TNtot)\ .\ V_D}.\ 100 \tag{1}$$

$$\%\ TN\ (distillate) = \frac{\rho_{D(TN)}\ .\ V_D}{\rho_T(TN)\ .\ V_T\ +\ \rho_D(TN)\ .\ V_D}.\ 100 \tag{2}$$

where ρ_T (TN) and ρ_D (TN) represent volumetric TN concentrations in mg/L in thickened LPD and distillate; V_T and V_D represent volumes (L) of thickened LPD and distillate. Percentage distribution of TAN was calculated as:

$$\%\ TAN\ (thickened\ LPD) = \frac{\rho_T(TAN)\ .\ V_T}{\rho_{T(TAN)}\ .\ V_T\ +\ \rho_D(TAN)\ .\ V_D}.\ 100 \tag{3}$$

$$\%\ TAN\ (distillate) = \frac{\rho_{D(TAN)}\ .\ V_D}{\rho_T(TAN)\ .\ V_T\ +\ \rho_D(TAN)\ .\ V_D}.\ 100 \tag{4}$$

where ρ_T (TAN) and ρ_D (TAN) represent volumetric TAN concentrations in mg/L measured in thickened LPD and distillate.

3. Results and discussion

3.1. *Basic characteristics of thickened nitrified LPD and its potential applications*

The experiments revealed that almost all nitrogen from the original samples of nitrified LPD was still present in thickened nitrified LPD, with an increase both in the TN volumetric concentration in thickened nitrified LPD and in the distillate/thickened nitrified LPD volume ratio, reaching a value of 17.9 g/L in the third series (Table 2).

$N{-}NO_3^-$ remained the dominant nitrogen form in thickened nitrified LPD. Mass concentration of TN in thickened nitrified LPD was 123–128 g/kg_{DM} depending on the series. Thus, nitrogen represented 12–13% of dry matter in all evaporation series. These values are similar to the TN mass concentration observed in nitrified LPD entering the process of thermal thickening and comparable to values usually observed in digestate (Botheju et al., 2010; Vaneeckhaute et al., 2017; Bolzonella et al., 2018; Shi et al., 2018).

Table 2

Basic characteristics of thickened nitrified LPD in three evaporation series.

Parameter (unit)	Evaporation series 1	Evaporation series 2	Evaporation series 3
pH (–)	6.4 ± 0.05	5.8 ± 0.1	5.8 ± 0.1
TS (g/L)	97.2 ± 5.25	120.3 ± 6.31	143.5 ± 8.1
$N{-}NO_3^-$ (mg/L)	11,500 ± 86	14,590 ± 285	16,850 ± 569
$N{-}NO_2^-$ (mg/L)	2.0 ± 0.1	1.0 ± 0.1	1.6 ± 0.6
TAN (mg/L)	15.4 ± 2.6	14.0 ± 1.5	14.2 ± 3.8
TN (g/L)	12.0 ± 0.10	15.4 ± 0.31	17.9 ± 0.62

At Evaporation series 3, 0.060% of TN from the nitrified LPD was transferred into the distillate during thermal thickening. This value was lower in Evaporation series 2 (0.048%) and Evaporation series 1 (0.035%). Under such conditions, over 99.9% of nitrogen contained originally in nitrified LPD was conserved in thickened nitrified LPD after vacuum evaporation in the three series. Overall, this indicates a very slight decrease of % TN (thickened LPD) with an increase in applied distillate/thickened LPD volume ratio.

The concentration of other nutrients in thickened nitrified LPD in each series is shown in Table S1. The results are similar to values reported in previous studies of fertilising potential of raw LPD and digestate (Vaneeckhaute et al., 2017; Bolzonella et al., 2018; Khan and Nordberg, 2019), with no significant differences between series. However, it is necessary to emphasise the amount of nutrients retained in thickened form after thermal thickening of nitrified LPD. For this reason, a slight increase in the volumetric concentration of nutrients was observed with the increase of distillate/thickened LPD volume ratio in each series of the experiment.

Different risk elements in the thickened nitrified LPD were also monitored to evaluate the environmental risks of thickened nitrified LPD as a fertiliser (Table S2). The results indicate values satisfying security standards from different countries (Siebert, 2008; British Standards Institution, 2014; The Ministry of Agriculture of the Czech Republic, 2017; European Parliament, 2019). However, the presence of risk elements depends on the chemical composition of original digestate, or more precisely on the quality of the substrate for biogas production at a given biogas plant (Al Seadi et al., 2013). As in the case of nutrients, the volumetric concentration of risk elements slightly increased with the distillate/thickened LPD volume ratio.

Based on its properties, it follows that thickened nitrified LPD is suitable as a complex concentrated fertiliser. However, experimental verification of the fertilising properties of thickened nitrified LPD including the test with plants is still required.

3.2. *Basic characteristics of distillate and its potential applications*

The concentration of TN in distillate reached very low levels (4.2–5.3 mg/L), with TAN as the nitrogen form exhibiting the highest concentration (Table 3). Although TAN concentration was higher than in the permeate obtained by membrane distillation (Khan and Nordberg, 2018), the values are still satisfactory. Distillate/thickened LPD volume ratio did not significantly influence TN concentration in the distillate. Low values of EC at 0.01–0.03 mS/cm confirm the very low content of salts in the distillate across all the evaporation series, with values

Table 3

Basic parameters of distillate.

Parameter (units)	Evaporation series 1	Evaporation series 2	Evaporation series 3
pH (–)	8.3 ± 0.10	8.0 ± 0.1	7.9 ± 0.1
EC (mS/m)	0.03 ± 0.007	0.02 ± 0.002	0.01 ± 0.003
$N{-}NO_3^-$ (mg/L)	1.1 ± 0.3	1.9 ± 0.4	1.2 ± 0.6
$N{-}NO_2^-$ (mg/L)	0.03 ± 0.01	0.03 ± 0.02	0.06 ± 0.03
TAN (mg/L)	3.1 ± 0.5	3.0 ± 0.6	4.0 ± 1.3
TN (mg/L)	4.2 ± 0.2	4.9 ± 1.0	5.3 ± 0.7
COD (mg/L)	180 ± 4	110 ± 3	100 ± 3

comparable to the permeate produced by a more complex membrane distillation process (Khan and Nordberg, 2018, 2019).

The properties of distillate suggest its potential use as technological water for the improvement of DM content in the substrate for anaerobic biogas production. Such technological water prevents the inhibition of anaerobic digestion by high ammonia levels, and thus the risk of raw LPD application for this purpose (Hu et al., 2014; Drosg et al., 2015). Moreover, the problem of returning inert material into the digester (Lindmark et al., 2011; Khan and Nordberg, 2019) may be also eliminated by the recirculation of distillate instead of raw LPD into the digester.

3.2.1. Transfer of TAN into distillate and its resulting risks

Although TAN concentration in distillate was much lower than through thermal thickening of raw LPD (Bonmatí and Flotats, 2003; Panvini et al., 2009; Chiumenti et al., 2013), a significant fraction of TAN originally contained in nitrified LPD was transferred into the distillate. As the consequence, the resulting fraction of TAN contained in the distillate was relatively high (16.8–36.4%), and increased with the distillate/thickened nitrified LPD volume ratio (Fig. 1).

Overall, the intensity of TAN transfer into distillate is determined by the pH value during thermal thickening of raw digestate as well as raw LPD (Bonmatí and Flotats, 2003; Panvini et al., 2009; Chiumenti et al., 2013). The results indicate the same trend during thermal thickening of nitrified LPD despite the much lower TAN concentration compared to raw LPD. Panvini et al. (2009) obtained 15% of TAN in distillate at pH 6.5 during the thermal thickening of raw digestate, which is relatively close to results of this study (especially Evaporation series 1). However, at pH value reaching 7.5–7.9 for example, even 78.1% of TAN present originally in raw LPD could be transferred into the distillate during thermal thickening (Chiumenti et al., 2013). These findings indicate a risk of relatively intensive transfer of nitrogen into the distillate in cases where TAN concentration in nitrified LPD exposed to thickening process is significantly higher than presented in this study, especially at higher distillate/thickened LPD volume ratios.

3.2.2. Possible strategy for minimisation of TAN transfer into distillate

It is essential to keep TAN concentration in nitrified LPD exposed to thermal thickening at the low values and/or to keep pH value at low levels below ca. 6.5 (Panvini et al., 2009), in order to minimise the amount of nitrogen transferred into the distillate. This can be achieved through careful control of the nitrification reactor operation ensuring pre-treatment of LPD before thermal thickening where the pH value and TAN concentration in the nitrification reactor treating the LPD before thermal thickening should be monitored, and if necessary, also controlled depending on the requirements for the maximum admissible TAN concentration in the distillate (Svehla et al., 2017).

Alternatively, addition of acid directly to nitrified LPD containing a higher concentration of TAN at a higher pH values can be performed as in the case of thermal thickening of raw LPD or digestate (Chiumenti et al., 2013; Panvini et al., 2009; Bonmatí and Flotats, 2003). In such cases, minimal consumption of acid is expected due to the decrease in the pH (or more precisely in alkalinity) of LPD during its nitrification (Botheju et al., 2010; Svehla et al., 2017). This assumption was experimentally confirmed by described tests. For example, the consumption of strong monohydric acid was 7.6 times lower when nitrified LPD was acidified to pH 4 compared to acidification of raw LPD at the same pH. The trend of acid consumption during pH improvement of nitrified and raw LPD used for this experiment is presented in Fig. 2. The curve trajectory is dependent on buffering capacity which is derived from the chemical composition of the sample, or more precisely, from the concentration of particular pH buffering substances. Thus, in accordance with Fig. 2, a non-linear trajectory of the curve is typical of water environments containing high concentrations of $(HCO_3)^-$, VFAs, TAN, $(HPO_4)^{2-}$ and other compounds such as LPD (Van Vooren et al., 2001).

The strategy of acid addition before thermal thickening of nitrified LPD may be particularly useful during periods characterised by limited efficiency of the nitrification reactor.

3.2.3. Organic compounds in distillate

The value of COD in the distillate ranging from 100 (Evaporation series 3) to 180 mg/L (Evaporation series 1) indicates a risk of transport of part of organic compounds from nitrified LPD into the distillate. The decrease in COD with the increase of the distillate/thickened nitrified

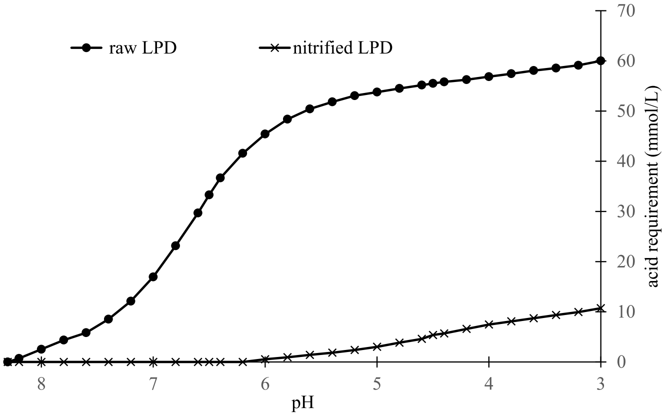

Fig. 2. Acid consumption during acidification of raw LPD and nitrified LPD.

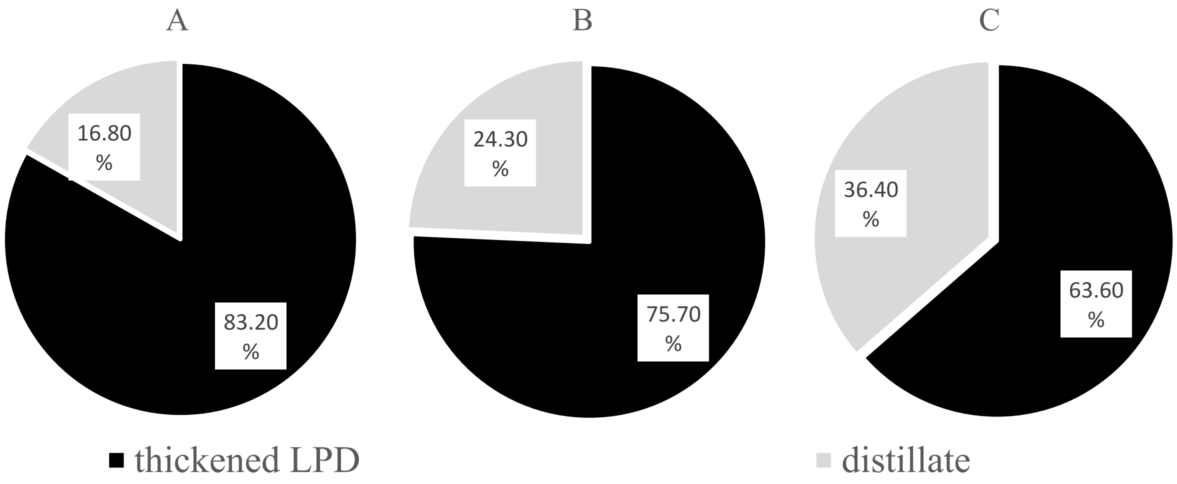

Fig. 1. Distribution of TAN between products of thermal thickening of nitrified LPD (A: Evaporation series 1; B: Evaporation series 2; C: Evaporation series 3).

LPD volume ratio indicates that only a limited amount of volatile organic compounds under selected experimental conditions was present in the sample. These compounds were transferred into different amounts of distillate over the three experimental series.

Presence of VFAs in the distillate may be partially responsible for increased COD values in this stream as suggested by Palatsi et al. (2005), who observed an increase of COD in distillate with increasing VFAs concentration in raw digestate exposed to thermal thickening. However, Palatsi et al. (2005) observed COD values of 300–800 mg/L based on the actual VFAs concentration in digestate. The degradation of VFAs and other biodegradable organic compounds in nitrification reactor pre-treating the LPD before vacuum evaporation seems to be responsible for significantly lower COD values in distillate in these experiments compared to Palatsi et al. (2005). COD reported values are also lower than in permeate derived from membrane distillation applied in LPD treatment (300–500 mg/L; Khan and Nordberg, 2018).

If we compare the concentration of particular VFAs in the original nitrified LPD, thickened nitrified LPD and distillate from Evaporation series 1, it follows that certain transformations of VFAs may have taken place during thermal thickening of nitrified LPD. For example, propionic acid was not detected in original nitrified LPD, while its concentration in thickened nitrified LPD was 43 mg/L. Similarly, butyric acid was observed in thickened nitrified LPD but not detected in original nitrified LPD (Table S3). The production of selected VFAs at an elevated temperature was repeatedly confirmed by previous studies of thermal hydrolysis of organic materials performed to enhance their biological degradability (Bjerg-Nielsen et al., 2018; Yang et al., 2019; Zhang et al., 2019). Overall, results of this study suggest that a fraction of VFAs can be transferred into the distillate during the process of thermal thickening of nitrified LPD, with a higher isobutyric acid concentration observed in the distillate compared to the thickened nitrified LPD. Finally, while increased COD values in the distillate can limit some of its applications, the use of distillate to dilute the substrate for biogas production is not affected by its recorded organic matter content.

3.3. Benefits of LPD treatment by nitrification and subsequent thermal thickening compared to thermal thickening of raw LPD and other LPD treatment processes

Different technologies for LPD treatment based for example on ammonia stripping (and subsequent acidic air scrubber application), struvite precipitation, ammonia and phosphorus sorption, application of vapour membrane contactor (and potential subsequent phytoremediation), eventually algae biomass production and harvest were previously introduced (Vaneeckhaute et al., 2017; Bolzonella et al., 2018; Monfet et al., 2018; Shi et al., 2018; Ortakci and YesilTugtas, 2019; Vondra et al., 2019). These methods can solve the problem of nitrogen (and/or phosphorus) management during digestate handling. However, all methods above are selective as they conserve nitrogen and/or phosphorus in separate streams. Most other nutrients remain in the resting (waste) material where their potential utilisation is questionable.

Thermal thickening together with membrane filtration seem to be the only methods able to channel all nutrients contained in original LPD into a single concentrated stream. However, membrane filtration shows excessive operational costs, problems related with high TS content in LPD and other technical problems (Vaneeckhaute et al., 2017; Khan and Nordberg, 2019). Although the main problems related to the application of membrane process to LPD treatment may be solved by the application of specific advanced technologies such as the membrane distillation system (Khan and Nordberg, 2018, 2019), the treatment seems to be still relatively complicated e.g. due to the necessity to acidify LPD to extremely low pH values (around 2–3).

During thermal thickening of raw LPD (or digestate), the application of chemicals is necessary for satisfactory nitrogen management. Chiumenti et al. (2013) found that the percentage of nitrogen transferred from raw LPD (pH in the range 7.5–7.9) into distillate was as high as 78.1%. To reduce nitrogen loss to 0.8%, it was necessary to reduce the pH to 3.5. Panvini et al. (2009) observed that with an initial pH of 8.4 around 65% of the nitrogen was transferred from digestate into distillate, whereas 15% was transferred when the initial pH of the digestate was adjusted to 6.5. Bonmatí and Flotats (2003) evaluated the influence of initial pH on the intensity of nitrogen transfer to the distillate, and observed optimal results with a pH of 4. Under such conditions, 0.5% of nitrogen was contained in the distillate. In the experiment with nitrified LPD described in this paper, TN loss was even lower despite no chemical improvement of treated material, which is relevant from the economic perspective.

The method of nitrification and subsequent thermal thickening of nitrified LPD has additional benefits. Thanks to the stability of nitrate, nitrogen losses during the storage of nitrified LPD before its thermal thickening, storage of thickened nitrified LPD, and during its application to soil are minimised (Svehla et al., 2020). Such losses are significant in the case of raw LPD handling (Whelan et al., 2010; Nkoa, 2014; Perazzolo et al., 2017; Nicholson et al., 2017; Svehla et al., 2020).

Taking into consideration all the evidence, the conclusion is that the technology based on the combination of biological nitrification of LPD and subsequent thermal thickening of nitrified LPD represents a valid alternative to existing processes.

Properly controlled nitrification seems to be a relatively universal pre-treatment of LPD method that could be effectively combined both with thermal thickening and also with other subsequent treatment processes. From this perspective, especially advanced nitrogen removal processes such as partial nitritation/anammox (Lotti et al., 2019) or nitrate reduction over indium-decorated palladium nanoparticle catalysts (Guo et al., 2018) could be combined with nitrification of LPD whenever it is handled as wastewater. Simultaneously, nitrification of LPD could be also used to produce nitrate/nitrite useful as the electron acceptor for autotrophic denitrification within biogas upgrading by hydrogen-sulphide scrubbing and bio-oxidation of captured sulphides (Pokorna et al., 2015).

3.4. Potential technical and economic problems

Thermal thickening of LPD involves some difficulties. In our experiment, the distillate/thickened LPD volume ratio varied from 1.0 (series 1) to 2.0 (series 3). TS concentration and the resulting dry matter content in thickened nitrified LPD reached relatively high values of 144 g/L, complicating its pumping and transportation (Al Seadi et al., 2013). TS content in original LPD seems to be the main factor determining the applicable distillate/thickened LPD volume ratio at each concrete BGP facility. On the other hand, Chiumenti et al. (2013) demonstrated that the production of thickened digestate of solid matter nature characterised by a TS content of 59% represents a suitable alternative for digestate management. Another possible downside is the high heat energy requirements of thermal thickening of nitrified LPD. However, BGPs usually uses only 20–40% of the heat produced in cogeneration units, with the remaining being wasted (Vondra et al., 2016, 2018bib_Vondra_et_al_2016bib_Vondra_et_al_2018). Therefore, thermal thickening of nitrified LPD can be performed with relatively low operational costs while at the same time re-using waste heat to provide BGPs with a productivity boost, as previously reported for the thickening of raw digestate (Al Seadi et al., 2013; Guercini et al., 2014; Fechter and Kraume, 2016; Vondra et al., 2016).

It follows that actual conditions prevailing at selected BGPs must be taken into account before the installation and operation of the proposed system of LPD nitrification and subsequent thermal thickening of nitrified LPD. In contrast to the processes proposed in previous studies, the method presented in this study has only been investigated at a laboratory scale. For more objective economical and technical evaluations and thorough comparison with other processes, the installation and long-term operation of a pilot plant unit performing nitrification/thermal thickening process at a real BGP is required. In particular, the biological nitrification process should be studied with an emphasis on energy demands, investment and operational costs.

4. Conclusions

The reported experiments demonstrated that the two-stage treatment of LPD consisting of nitrification of raw LPD and subsequent thermal thickening of nitrified LPD may yield a valuable complex fertiliser. Another outcome is the production of technological water of relatively high quality, which may be applied to DM content optimisation of the substrate entering anaerobic digestion. Minimisation of TAN concentration and/or pH optimisation in nitrified LPD entering the process of thermal thickening seems to be essential for the minimisation of TN content in the distillate. Transfer of VFAs and other volatile organic compounds into distillate may increase COD values in this stream. Despite the potential risks, with a satisfactory operation of the nitrification stage the quality of distillate produced by thermal thickening of nitrified LPD may be significantly higher compared to thermal thickening of raw LPD.

CRediT authorship contribution statement

Pavel Svehla: Methodology, Conceptualization, Writing - original draft, Data curation. **Liz Mabel Vargas Caceres:** Formal analysis. **Pavel Michal:** Formal analysis, Writing - review & editing. **Pavel Tlustos:** Supervision.

Declaration of competing interest

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.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jenvman.2020.111250.

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