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Research article

Two-stage separation and acidification of pig slurry – Nutrient separation efficiency and agronomical implications

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ABSTRACT

Separation of slurries can facilitate the nutrient management on farms through nutrient partitioning between the liquid and the solid fraction. The distribution of nutrients in the slurry fractions depends largely on the type of separator used. The current study assessed the separation efficiency of a two-step separation treatment of pig slurry including in-series a screw press and a centrifuge followed by acidification (to pH 5.9) of the final liquid effluent. The system concentrated 73.8% of the slurry's Phosphorus (P) content, 52.6% of Total solids (TS) and 14.4% of total Nitrogen to the solid fraction. The apparent N recovery from ryegrass fertilized with the raw slurry and non-acidified liquid fractions was not decreased by the separation treatment. The acidified liquid fraction showed 28% and 9% higher apparent N recovery compared to the raw slurry and the non-acidified liquid effluent from the centrifuge respectively. The biochemical methane production potential (B_o) of the acidified liquid fraction was reduced by 50% and 25%, compared to the non-acidified counterpart and the raw slurry, respectively. The results highlight the potential of a double separation system coupled with acidification of the liquid fraction, to extract P into a solid fraction which can be transported outside the farm, and to increase N utilization from the liquid fraction when this is used as organic fertiliser on or nearby the farm. The study further highlights the potential to reduce CH_4 emissions from slurry storage after mechanical separation and acidification of the liquid slurry fraction.

1. Introduction

In Europe alone, 1,300 million tons of manure are produced annually, and their utilization in agriculture can facilitate the efforts towards reduction of producers' dependency on mineral fertilizers and sustain soil quality (Scarlat et al., 2018). Nevertheless, in modern, high density livestock systems, manure often accumulates in specific regions which strongly associates with environmental risks including, ecosystem eutrophication, soil acidification and global warming (Rojas-Downing et al., 2017).

The livestock sector is responsible for 78% of Europe's ammonia (NH_3) emissions and 14% of greenhouse gas (GHG) emissions (Gerber et al., 2013). Moreover, previous reports indicated that poorly managed manures contribute with 50% of the nitrogen (N) and 70% of the phosphorus (P) losses accountable for water quality degradation (Leip et al., 2015). This is specifically important for eutrophicated areas, such as the Baltic Sea region, where nutrient surpluses in agriculture have been identified as the main contributor of non-source pollution leading to the eutrophication of the Baltic Sea (Svanbäck et al., 2019).

Animal slurries often show a low N to P stoichiometric ratio therefore, their utilization as fertilizers, to meet crops N demands, often applies P in excess of crop requirements increasing the risk of P escaping to water bodies. Phosphorus is a non-renewable source and the largest proportion of phosphorus mineral reserves are found in only a few countries namely Morocco, China and the United States. In this context, the EU has placed phosphorus in the list of critical raw materials and urge its efficient use and recycling (European Commission, 2017). Nevertheless, the high-water content of liquid manures is among the main bottlenecks for efficient recycling of nutrients between areas with nutrient excesses and areas with respective deficiencies. Mechanical separation of slurries has been proposed as an adequate first step in manure management plants which relies on the partitioning of slurry in a liquid stream, rich in ammonium nitrogen (NH_4–N) and potassium (K), and a solid fraction rich in organic matter, P and relatively rich in nitrogen (Hjorth et al., 2010). The concentration of P and organic matter in the solid fraction, enhances nutrient management in the farm and

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facilitates a more feasible exportation of excessive nutrients to areas with respected deficiencies. The respectively depleted liquid fraction can be stored until further used, as a more nutrient balanced organic fertilizers, in crop fields close to the farm. The lower total solids (TS) concentration in the separated liquid fraction, compared to the initial slurry, decreases hauling costs related to slurry pumping and reduces NH_3 emissions during the application stage due to a more rapid infiltration in soil (Guilayn et al., 2018). The distribution of moisture, organic matter and nutrient content between the solid and the liquid fraction varies widely between different slurry separators and largely depends on the type of the separation system and the characteristics of the influent material (Tambone et al., 2017). The selection of the separation technology or the combination of technologies relies on the characteristics of the initial slurry and the site-specific targets of the manure management plan.

Acidification of slurries has been identified as an adequate method to reduce NH_3 emissions from the slurry management chain which also can modify slurry characteristics with direct impact on the fertilizer value of the slurry (Fangueiro et al., 2017). A range of inorganic and organic acids have been proposed for the acidification of slurries. Nevertheless, use of inorganic acids has been demonstrated to provide more stable pH conditions in the slurry, and higher efficiency to suppress NH_3 emissions from slurry storage compared to the respective use of organic acids (Regueiro et al., 2016a). Among the several acids proposed for slurry acidification, the inexpensive sulfuric acid (H_2SO_4) is mainly utilized to reduce slurries' pH between 5.5 and 6.0 (Fangueiro et al., 2015).

While extensive research has been carried out to assess the separation efficiency of different technologies in lab or semi-scale conditions, only a few reports concern full scale separation systems utilizing simultaneously more than one separation technologies. Similarly, while acidification has drawn attention as a treatment preceding mechanical separation, few data are available on acidification of solely the liquid effluent, in large scale installations. For the rational selection of slurry treatments, evidence-based results on separation systems efficiency and fertilizing value of products from large scale installations are necessary for potential users and policy makers.

There were two main objectives of this study, 1) to determine the TS and nutrient separation efficiency of a two-step pig slurry separation system and 2) to assess the effect of acidification as a strategy for further treatment of the liquid effluent. Agronomic aspects related to the N fertilizing value of the on-farm produced liquid fractions and the biochemical methane potential of slurry and the different slurry products was also evaluated.

2. Materials and methods

2.1. Slurry treatment plant

The investigated farm was located in Przybkowo, Poland. At the time of the study, in 2018, there were approximately 30,000 pigs in different growth stages, which produced approx. 60,000 m^3 slurry per year. The farm had been operating continuously for the past 40 years and is surrounded by 900 ha own agricultural land consisting of sandy soils with poor structure. Until 2016, the bulk slurry was stored in an open storage basin until utilization as organic fertilizer on the farms' own land, as is common practice in the region.

In 2016, a new manure treatment plant was installed, consisted of an in-series double separation scheme coupled with acidification of the final liquid effluent (Fig. 1). Slurry from the individual animal houses was collected in a balance tank where it was constantly stirred with a mechanical mixer. The mixed raw slurry (RS) before separation, underwent maceration where potential impurities curried in the slurry (e.g. wood pieces, stones, etc.) were grinded, to avoid risks of clogging and of mechanical failure in the subsequent separators. The first separation step of the pig slurry included a screw press (PSS 3.2–520, FAN Separator, GmbH) with 0.5 mm screen openings. The produced liquid fraction from the screw press (SL) underwent a second separation step in a decanter centrifuge (Aldec 45, Alfa Laval). The liquid effluent from slurry's centrifugation (CL) was collected in a tank where concentrated sulfuric acid was added to reduce the liquid's pH close to 6. The acidified liquid (ACL) was then pumped to covered lagoons until used as organic fertilizer for crop production. The solids produced from the screw press (SS) and from the centrifuge (CS) were collected in containers and exported daily to a proximate (30 km) anaerobic digestion plant.

2.2. Monitoring sampling

Liquid and solid samples from the manure treatment plant were collected from the whole slurry management chain, twice per month for a period of 5 months (February to June 2018) (Fig. 1). The acidified liquid fraction was collected after mixing the liquid content of the acidification tank using the tank's mixer. Respective samples were collected from the center of the middle layer of the acidified liquid, with a liquid sampler. In each sampling date, three sub-sample (1.5 l) from every sampling point were collected in hermetically closed plastic containers every 3 h for 6 h, resulting in nine sub-samples from each sampling point. At the end of the sampling, the respected sub-samples were mixed and three representative aliquots (1.5 l for liquid samples, 500 g for solid samples) were collected and kept frozen (−18 °C) until analysis. The volume of the raw slurry processed and of the liquid effluents produced after each slurry treatment were estimated based on installed flowmeters and tank probes. The mass of solids produced during each sampling period was estimated by weighing the solids holding containers at the end of each sampling event.

2.3. Greenhouse experiment

The nitrogen plant availability from the unprocessed slurry and the derived liquid fractions was assessed in a greenhouse experiment with ryegrass (*Lolium multiflorum*) for four months. The soil used had a sandy loam texture (6% clay, 17% silt and 75% sand) with pH (H_2O) 6.2, 2.5% organic matter, 5.5 mg kg^{-1} NH_4–N, 17.3 mg kg^{-1} NO_3–N, 157 and 100

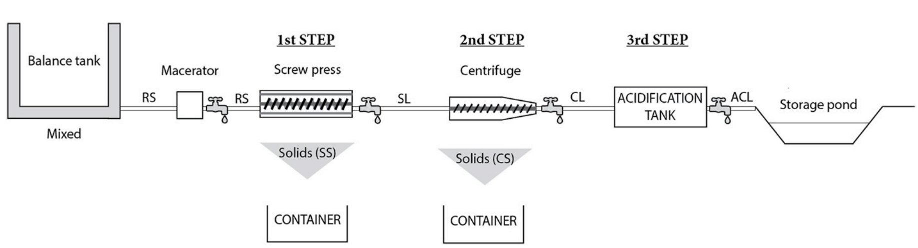

Fig. 1. Schematic illustration of the pig slurry treatment scheme. Pig slurry from the balance tank is pumped to the separation plant after homogenization in the macerator and undergoes three treatment steps. 1st step, raw slurry is separated in a solid fraction (SS), which is collected in containers, and a liquid fraction (SL) which is further processed. 2nd step, SL is centrifuged and the produced solids (CS) are collected in containers while the produced liquid fraction (CL) is further treated in the 3rd step which consists of an acidification tank where pH of the liquid is reduced close to 5.9. The acidified liquid (ACL) is then pumped to the storage pond until used as organic fertilizer. Sampling points are indicated by faucet symbols.

mg kg^{-1} lactate extractable K and P respectively. The soil was sieved through a 5 mm mesh and pre-incubated in the greenhouse for three weeks. The moisture content of the soil was maintained to 50% of its water holding capacity by addition of distilled water.

After the end of the pre-incubation period, 3 kg of soil (dry weight) were mixed with either unprocessed slurry (RS), liquid fraction after pressurized filtration of raw slurry (SL), liquid effluent after centrifugation of SL (CL) or with acidified CL (ACL) and placed in 4 l pots. The addition of slurry and derived liquid fractions aimed to reach a total nitrogen input of 170 kg ha^{-1}, which is the upper limit of yearly permissible agricultural utilization of organic fertilizers (Table 5). Pots without any N and P inputs served as control treatment. All treatments received 300 mg pot^{-1} K (KCl) as basal fertilization and replicated four times. The soil was gently packed to 1.49 g cm^{-3} and distilled water was added to raise the moisture content to 65% of WHC. Stable moisture levels in all pots was secured by frequent irrigation with distilled water, throughout the experiment.

A first pre-cut was performed 10 days after plant emergence to standardize the starting point and the cut biomass was left in the respected pots. Three main cuts were performed at 30, 60 and 90 days after the first pre-cut. Ryegrass was cut 4 cm above the soil surface and harvested for estimation of aboveground biomass production and N content.

2.4. Analytical methods

2.4.1. Slurry and slurry fractions

Frozen samples of slurry and slurry fractions were thawed at room temperature in closed containers for 24h. Total solids content (TS) was estimated after drying of samples to constant weight at 80 °C. Volatile solids (VS) were determined by loss on ignition at 550 °C for 24h. Electrical conductivity and pH were measure with a dual pH/conductivity electrode directly immersed in the liquid samples or in distilled water solution of the solids (1:10, solids:water, v/w). Ammonium (NH_4–N) and nitrate (NO_3–N) nitrogen content of the samples were determined after extraction with 1 M KCl and filtration of extracts by flow injection analysis. Carbon (C) and total N contents of dried samples were estimated on a CN auto-analyzer (Carlo Erba NA2000, Milan). Phosphorus and all micronutrients were determined in dry samples with ICP-OES in chemically digested samples. Nitric acid (HNO_3) and hydrogen peroxide (H_2O_2) were utilized as digestion agents to improve the dissolution of the solids (Hansen et al., 2013).

2.4.2. Biochemical methane potential

The biochemical methane potential (B_o) of slurry and slurry fractions was determined under mesophilic conditions (38 °C) in triplicate batches using 0.5 l glass bottles. The test was performed in an AMPTSII system (Bioprocess Control Sweden AB, Lund) and the inoculum was prepared by co-digested pig manure- and plant residues-based digestate. The organic load for each treatment was 6.0 g VS/L equal to approximately 13–267 g of substrate per bottle and the ratio of the substrate to the inoculum was 0.1. Methane production was monitored on a daily basis until gas production was levelled off and stabilized, and the dried biogas volume was converted from mesophilic conditions to standard conditions i.e. air pressure 1013.25 hPa and temperature 273.15 K.

2.4.3. Plant biomass

Ryegrass yields from the different treatments were determined after biomass oven-drying at 45 °C for 96h. The dry biomass was weighted, ball-milled and analyzed for total N content by the Kjeldahl method (Bremner, 1960).

2.5. Calculations

The widely used, simple separation index was adopted to determine the specific efficiency for each separator, to concentrate nutrients to the solid fraction (Et) (Hjorth et al., 2010):

$$Et_x = m_x solid * 100 / m_x slurry \quad (1)$$

where m_x represents the mass of dry matter, volatile solids or any specific compound. In the current study, the simple separation index was chosen, in accordance with Burton, (2007) who suggested this as an efficient method to establish the efficiency of the separators in the current context.

To estimate the nutrient depuration of the raw slurry after the separation treatments, on a mass basis, the system's removal efficiency was calculated as follows:

$$\text{System removal}(\%) = ((\text{system load} - \text{system effluent}) / \text{system load}) * 100 \quad (2)$$

Nitrogen content (NU) in plant material was calculated by multiplying shoot dry matter yields (g DM pot^{-1}) with N concentration (mg N g^{-1} DM) of the harvested biomass. Apparent N recovery (ANR) was calculated as:

$$\text{Apparent N recovery}(ANR) = (\text{NUtreatment} - \text{NUcontrol}) / \text{total N applied} \quad (3)$$

2.6. Statistics

Results from the monitoring samplings were analyzed using one-way analysis of variance (ANOVA) to assess the differences in slurry and slurry fraction characteristics. A two-way ANOVA was performed to assess the effect of the liquid slurries and biomass cut harvest on ryegrass DM yield and apparent N recovery. The Levene's test was utilized to assess the assumption of equal variances of the different groups. The significance of the differences between treatments within each biomass cut and cumulatively was estimated with the Tukey's post-hoc test (HSD). The statistical analysis was performed with SPSS 24.0 software package (IBM Inc., Chicago IL).

3. Results and discussion

3.1. Characteristics of raw slurry

The composition of the raw pig slurry (RS) during the sampling period is presented in Table 1, as the mean of ten different sampling events performed from February to June 2018. In the current study, the slurry showed mean value of 43.6 g kg^{-1} TS content and was characterized as high strength wastewater (González-Fernández, 2008). The TS content of the slurry as well as the respective N and P concentrations did not fluctuate considerably during the monitoring period (not shown), indicating standardized animal production and slurry management practices in-barn.

The average total N content of RS was 3.82 g kg^{-1} slurry (wet weight basis: w.w) and the inorganic fraction of N was in the form of ammonium nitrogen (NH_4–N), accounting for 63% of total N (Table 1). This represents a typical value of the proportion of inorganic N to total N content in slurries since close to 60% of N excreted by pigs are via urine which is rapidly hydrolyzed to inorganic nitrogen (Sommer et al., 2013). The total P content of RS was 0.89 g kg^{-1} (w.w) and was slightly alkaline with mean pH value 7.2. Makara and Kowalski (2018) reported considerably lower total N and total P concentration values, in five different pig slurries in Poland, ranging from 1.0 to 2.4 mg kg^{-1} (w.w) and 0.08–0.31 mg kg^{-1} (w.w) respectively. Discrepancies in slurry characteristics between farms may be attributed to animal feed composition, physiological state of the animals and slurry management practices in-barn (Riaño et al., 2015). The copper (Cu) and zinc (Zn) content (Table 1) of the raw slurry showed mean concentrations of 20.1 mg kg^{-1} TS and 87.4 mg kg^{-1} TS respectively, which agree favorably with values reported elsewhere for similar types of slurry (Cattaneo et al., 2019).

Table 1
Mean characteristics of raw slurry (RS) and of derived liquid fractions after screw pressurization (SL), centrifugation (CL) and acidification (ACL) during the monitoring period (February–June). Standard deviation is shown in brackets (n = 10). Within each line, values followed by different letter are significantly different ($p < 0.05$) according to Tukey test (HSD).

Characteristics	Raw slurry (RS)	Screw press liquid (SL)	Centrifuge liquid (CL)	Acidified centrifuge liquid (ACL)
		$g\ kg^{-1}$ (±sd)		
TS*	43.6 (4.9)^a	32.1(1.7)^b	20.2(1.1)^c	22.7(1.0)^c
VS*	33.5 (1.4)^a	23.0(1.8)^b	14.1(2.1)^c	14.9(1.7)^c
Total N	3.82 (0.11)^a	3.71(0.05)^a	3.30(0.09)^b	3.60(0.16)^a
NH_4–N	2.38 (0.14)^a	2.37(0.02)^a	2.24(0.05)^a	2.44(0.06)^a
C_{tot}	18.07 (2.3)^a	13.24(1.4)^b	8.58(0.3)^c	7.77(0.1)^d
Total P	0.89 (0.11)^a	0.8(0.12)^a	0.25(0.04)^b	0.24(0.02)^b
K	1.51 (0.22)^a	1.58(0.09)^a	1.60(0.10)^a	1.50(0.11)^a
Mg	0.53 (0.10)^a	0.48(0.09)^a	0.10(0.03)^b	0.10(0.00)^b
Ca	0.93 (0.17)^a	0.84(0.10)^a	0.54(0.04)^b	0.61(0.05)^b
Na	0.39 (0.08)^a	0.41(0.05)^a	0.42(0.05)^a	0.41(0.04)^a
S	0.40 (0.05)^b	0.38(0.03)^b	0.30(0.04)^b	1.21(0.09)^a
		$mg\ kg^{-1}$ TS (±sd)		
Cu	20.1 (2.9)^a	17.8(1.0)^a	13.5(1.5)^b	14.3(2.2)^b
Fe	91.3 (6.9)^a	75.2(9.8)^a	37.3(7.2)^b	39.7(6.2)^b
Mn	29.0 (0.1)^a	26.7(1.0)^a	10.4(1.0)^b	7.63(2.2)^b
Zn	87.4 (18.6)^a	85.5(19.6)^a	59.6(12.4)^b	67.6(6.6)^b
		$m^3\ CH_4\ ton^{-1}$ VS (±sd)		
Bo*	418(59)^c	512(31)^b	618(22)^a	310(36)^d
pH	7.2(0.2)^a	7.2(0.2)^a	7.4(0.1)^a	5.9(0.1)^b
N_{tot}:P_{tot} ratio	4.29	4.64	13.20	15.00

*TS = Total solids, VS = volatile solids, B_0 = Maximum methane-producing capacity.

3.2. Slurry and liquid fractions characterization

The separation system of the farm gradually reduced the TS and nutrient contents of the slurry after each separation step (Table 1). During the monitoring period approximately 25.000 m^3 of RS were processed in the separation system of the farm which produced close to 22.000 m^3 of CL. The separation treatment of the slurry cumulatively reduced the TS content of RS by 54% in the resulting CL, which showed a mean TS content of 20.2 g kg $^{-1}$ (w.w). The total P content of RS was progressively reduced during the separation treatment to 0.80 g kg^{-1} (w.w) in SL and 0.25 g kg^{-1} (w.w) in CL representing an overall concentration reduction of 72%. The liquid effluent from the centrifuge showed a mean total N concentration of 3.30 g kg^{-1} (w.w), approximately 24% and 21% lower compared to the respective concentration, of RS and of the liquid effluent from the screw press. Higher nutrient concentration reduction values, between initial slurry and liquid effluent, have been reported by Ledda et al. (2013) in similar experimental setup. These authors reported system's reduction values in pig manure based digestate ranging from 68% for TS to 34% and 91% for total N and total P respectively. However, these results were obtained after the addition of flocculants to the digestate which has been shown to promote nutrient removal from the liquid streams (Popovic et al., 2012). The total N:total P ratio of the initial slurry was notably increased from 4.29 in RS to 4.64 in SL and 13.20 in CL due to the removal of P from the liquid stream of the slurry into the solid fraction (Table 1). Copper and Zn concentrations in CL were reduced by 34% and 31% compared to the initial raw slurry. Comparable results were obtained from Kumaragamage et al. (2016) who further highlights the potential to reduce the risk of heavy metal soil contamination after the application of separated liquid slurry as organic fertilizer (Table 1).

As expected, acidification of CL, with sulfuric acid, sharply reduced the pH in ACL from 7.4 to 5.9 and concurrently the sulfur (S) content of ACL was increased, compared to CL, due to the addition of sulfuric acid. The S content in ACL was close to 0.9 g kg^{-1} (w.w) higher to the respective concentration in CL indicating that approximately 3.7 kg of sulfuric acid were utilized to acidify 1 ton of CL. Acidification increased the total N content in ACL compared to CL nevertheless, the difference is largely attributed to the higher TS content of the former and was not statistically significant when considering the total N content on a dry weight basis.

3.3. Characterization of solids and system's efficiency

The efficiency of the screw press and of the centrifuge to transfer TS and nutrients to the respective solid fractions differed significantly and is well depicted in the nutrient concentration differences between the two solids (Table 2). Solids from the centrifuge showed a mean P concentration of 36.4 g kg^{-1} TS, compared to 12.9 g kg^{-1} TS in SS, i.e. 35% lower Table 2). Moreover, CS showed a mean total N content of 33.5 g kg^{-1} TS which was 35% higher than in solids from the screw press. This was in accordance with Peters and Jensen, (2011) who compared the nutrient content of differently separated solids from a wide range of slurries, with distinct characteristics. The NH_4–N content of the solids ranged from 35% to 25% of the total N in SS and CS respectively and was in agreement with Fangueiro et al. (2012) who report an NH_4–N:total N ratio of less than 50% for separated manure solids. Copper and Zn concentrations in SS were 45% and 46% lower respectively, compared to their counterparts from CS, where mean concentrations were 72.5 mg kg^{-1} TS for Cu and 32 mg kg^{-1} TS for Zn.

The separation index of the screw press and the centrifuge differed significantly ($p < 0.05$) (Table 3) with the former show peak values of 24.9% and 22.6% for C_{tot} and TS (Table 3). The high C separation efficiency of the screw press is well justified by its capacity to produce solids with high TS content (Hjorth et al., 2010). The separation index for total N and total P of the screw press ranged between 3.61% and 14.4% respectively (Table 3). A detrimental factor affecting the separation of nutrients from slurry is the solubility of the specific compounds and their

Table 2
Mean characteristics of the solid fractions produced after the screw press (SS) and the centrifuge (CS) during the monitoring period (February–June). Standard deviation is shown in brackets (n = 10).

Characteristics	Screw press solids (SS)	Centrifuge solids (CS)
	$g\ kg^{-1}$ TS (±sd)	
TS*	276(8.0)	254 (13.1)
VS*	243(13.2)	190(12.8)
Total N	22.5(1.3)	33.5(1.1)
NH_4–N	8.68(0.11)	9.82(0.12)
C_{tot}	453(18.0)	411(6.0)
Total P	12.9(0.9)	36.4(1.1)
K	6.05(0.32)	7.24(0.44)
	$mg\ kg^{-1}$ TS (±sd)	
Cu	39.9(4.6)	72.5(11.0)
Zn	14.6(2.2)	32.0(1.7)
	$m^3\ CH_4\ ton^{-1}$ VS (±sd)	
Bo*	418(59)	512(31)
pH	7.6(0.1)	7.8(0.1)

*TS = total solids, VS = volatile solids, B0 = Maximum methane-producing capacity.

Table 3
Overall and mean separation index (%) of each separator demonstrating the relative contribution on components concentration in the produced solid fractions.

Separation Index (%)[a]			
	Screw press	Centrifuge	Overall
TS	22.6	46.4	55.6
Total N	5.84	13.5	18.4
NH_4–N	3.61	6.20	9.5
C_{tot}	24.9	46.3	56.7
Total P	14.4	68.3	73.4
K	6.19	6.79	13.1
Cu	11.2	25.1	59.3
Zn	11.9	30.2	75.1

[a] Separation index (%) calculated according to equation (1).

association with slurry particles (Sommer et al., 2013). Therefore, the pore size of the sieve in the screw press has a significant effect on the quantity of particle associate nutrients that are concentrated in the solid fraction. In this study the pore size of the screw press was 0.5 mm and previous research indicates that only 22% of total N and 18% of total P are associated with particles >0.5 mm (Vanotti et al., 2009; Popovic et al., 2012) and the remaining amounts are either diluted or attached to smaller particles. Screw presses with smaller than 0.5 mm screen size may be able to increase the efficiency for nutrient removal from slurries nevertheless, these often demonstrate operational failures (Riaño et al., 2015).

In most cases, centrifuge outperformed the screw press in concentrating nutrients in the corresponding solid fraction (Table 3). Considering major nutrients in the slurry, the separation efficiency of the centrifuge peaked at 68.3% for total P. Previous research indicates that approximately 68% of the total P content in pig slurry is insoluble and associated with the organic matter of the slurry (Sommer et al., 2013). Compared to pressurized filtration, centrifugation is more efficient in transferring small particles (<20 μm) to the solid fraction thereby retaining particulate P in the produced solids (Peters and Jensen, 2011). Higher centrifuge P separation indexes, up to 91%, have been reported either in lab scale separation tests or in slurries with higher dry matter (Cocolo et al., 2012). Nevertheless, optimization of nutrient separation in the lab scale is more efficient and relates to higher retention times of the slurry in the separators. Moreover, difference in the TS content between slurries is expected to favor higher P separation efficiencies in slurries with higher TS (Fournel et al., 2018).

The relatively low separation index of the centrifuge for total N which showed a mean value of 13.4% can be mainly attributed to the high proportion of NH_4–N in the slurry. Coccolo et al., (2012) reported low total N recovery in the solid fraction, mainly because the dissolved NH_4–N remains suspended in the liquid fraction. However, the higher efficiency of centrifuge to separate total N compared to the screw press is largely attributed to its higher capacity to separate organic nitrogen associated with smaller particles.

The separation index values for Cu and Zn peaked in the case of the centrifuge at 25.1% and 30.3% respectively while the screw press demonstrated consistently lower respective values from 11.2% for Cu to 11.9% for Zn (Table 3). Similar with the case of P, both metals are mostly diluted or bounded at small sized particles which can easily pass through the 0.5 mm sieve (Popovic et al., 2012). Settling of smaller slurry particles in the centrifuges enhances the concentration of metals in the CS, therefore, increase the separation index of the treatment (Fournel et al., 2018).

During the five-months monitoring period, the slurry separation plant processed approximately 25.000 m^3 of raw slurry and produced close to 0.9 t of SS (w.w) and 1.4t CS (w.w). The system load for the reporting period was estimated on a wet mass basis according to eq. (2), to 1,087 t of TS, 95.5 t of total N, 58.7 t NH_4–N and 22.1 t total P (Table 4). The system's efficiency to remove nutrients from the liquid fraction of the pig slurry peaked for P where 73.8% of the initial P load was transferred to the solid fractions exported from the farm (Table 4). The acidified liquid effluent, which is stored in the farm until used for crop fertilization, showed reduced Cu and Zn mass loads by 66.1% and 63.3% respectively while the respective mass loads reduction for total N and NH_4–N was only 14.4% and 6.4% respectively. Comparable results were obtained, when calculating the system's mass balance based on the combination of the two separation indexes, represented by the overall separation index (Table 3), highlighting the precision of the measurements. Any small differences are largely attributed to the general difficulties to obtain precise mass balance estimation under field conditions.

Table 4
System load, effluent, and system efficiency at the farm during the monitoring period (February–June 2018).

	System load (raw slurry) kg	System effluent (after acidification) kg	System's removal efficiency %[a]
TS	1,087,000	515,000	52.6
Total N	95,500	81,666	14.4
NH_4–N	58,750	55,000	6.4
Total P	22,125	5,792	73.8
K	37,708	34,042	9.7
Cu	21.8	7.4	66.1
Zn	95.0	34.8	63.3

[a] System's removal efficiency (%) calculated according to equation (2).

3.4. Biochemical methane potential of liquid slurries

The maximum methane-producing capacity, estimated by the biochemical methane potential test, B_o, was 418 m^3 CH_4 t^{-1} VS in the raw slurry (Table 1). Available reports indicate that B_o values for pig slurry vary between 170 m^3 CH_4 t^{-1} VS and 642 m^3 CH_4 t^{-1} VS and the variability is reported to be largely attributed to differences in the animal feeding strategy adopted by different farms and to the physiological stage of the animals which strongly effect slurry characteristics (Jarret et al., 2011). The liquid fractions from the screw press and the centrifuge showed higher B_o values compared to raw slurry by 22% and 47% respectively. The progressive increases in B_o in the liquid effluents after each separation step is related partly to the reduction of recalcitrant carbon species (e.g. lignin) in the liquid fraction after solids removal through separation. The lignin content of organic waste has been negatively correlated with their biodegradability of the waste and therefore, their methane production potential (Rodriguez-Chiang et al., 2016). Sommer et al. (2014) reported that liquid effluents obtained from screw press and centrifuge had 16% and 84% lower lignin concentrations respectively. Acidification of CL decreased B_o by almost 50%, possibly due to an acidification with sulfuric acid induced inhibition of methanogenesis (Moset et al., 2012). Furthermore, Habtewold et al. (2018) suggested that low growth and activity of methanogens, especially *Methanosarcina* species, was responsible for 69%–84% lower CH_4 emissions from acidified liquid slurries compared to non-acidified. Methane emissions are directly related to the amount of slurry produced, the Bo of the slurry as well as its VS solids content (Supplementary material, S1). Therefore, slurry separation and acidification may significantly reduce potential CH_4 emissions from the separated liquids according to the IPCC (2006) estimation methodology, compared to the initial slurry, mainly through reduction in the organic matter content of the separated liquid fraction and the acidification induced reduction in the biochemical methane potential of the acidified liquid effluent (Supplemental material, S1).

3.5. Ryegrass yields and apparent nitrogen recovery

As expected, the different treatments, including unprocessed pig slurry or liquid fractions after separation of slurry, significantly

Table 5
Raw slurry and liquid products characteristics and nutrients applied to each pot through the different treatments. Treatments were applied on an equal total N basis (56.6 g kg-1 of soil) to pots containing 3.0 kg soil pot-1(d.w).

	Characteristics										Nutrient's applied		
Treatments	DM	pH	N_{tot}	NH_4–N	C_{tot}	P	K	Ca	NH_4–N:N_{tot}	C:N	N_{tot}	NH_4–N	P_{tot}
	(%)		mg g^{-1} (w.w)								mg pot^{-1} (w.w)		
Pig slurry (RS)	4.42	7.3	3.87	2.43	19.10	0.89	1.53	0.91	0.63	4.94	170	106.7	39.1
Screw press liquid (SL)	32.9	7.3	3.73	2.42	13.58	0.81	1.57	0.88	0.65	3.64	170	110.3	36.9
Centrifuge liquid (CL)	19.4	7.4	3.38	2.28	8.71	0.25	1.61	0.52	0.68	2.58	170	114.7	12.6
Acidified centrifuge liquid (ACL)	23.1	5.8	3.49	2.50	7.82	0.24	1.58	0.59	0.72	2.24	170	121.8	11.7

increased the total dry matter yields of ryegrass relative to the control, non-amended treatment (Fig. 2). Ryegrass yields obtained within each cut as well as cumulatively for all three cuts varied significantly with treatment ($p < 0.05$) and cut time ($p < 0.05$). In line with previous reports, the dry matter (DM) yields declined from the 1st to the 3rd cut, which is strongly related to the depletion of the inorganic N pool due to plant N uptake (Pantelopoulos et al., 2017).

Cumulative ryegrass DM yield in the different treatments followed the order, ACL (7.1 g pot^{-1}) <CL (6.4 g pot^{-1}) <SL (5.7 g pot^{-1}) = RS (5.7 g pot^{-1}) <Control (1.8 g pot^{-1}) (Fig. 2). Cumulative plant yields and ANR (44–45%) did not differ significantly between SL and RS which was in agreement with previous reported ANR range for raw and separated pig slurries (Sommer et al., 2013). The similar DM yields and ANR in RS and SL are likely attributed to similarities in the ryegrass availability of the N applied with those treatments. Differences in plant N availability between slurries have been previously correlated with respective differences in particle size distribution where, slurries with higher content in small size particle fractions show higher potential N mineralization (Fangueiro et al., 2010). However, the efficiency of the screw press to remove small size particles from pig slurry is low and Regueiro et al. (2016b) observed similar N release pattern between raw pig slurry and liquid fraction from pressurized filtration during a soil incubation study. This may explain the absence of significant differences between RS and SL in the current study.

Ryegrass fertilized with CL showed significantly ($p < 0.05$) higher cumulative DM yields and 22% higher ANR compared to the raw slurry (Figs. 2 and 3). Slurry separation through centrifugation has been shown to remove a substantial amount of C from the liquid which may have led to decreased N immobilization in the soil and subsequently result in higher N plant availability in the CL treatment (Table 1) (Fangueiro et al., 2012). Moreover, other studies have found that centrifuge effluents had lower lignin contents compared to the raw slurry which is strongly relates to reduced N immobilization after their application to soil (Sommer et al., 2014).

Ryegrass fertilized with ACL showed significantly ($p < 0.05$) higher cumulative dry matter yields and higher ANR (61%) compared to the rest of the treatments, indicating higher N availability due to acidification (Figs. 2 and 3). Plant N uptake relies on the mineral N content of the fertilizer, as well as the rate of mineralization of organic N after soil application of organic fertilizers. Since the NH_4–N:total N in ACL were similar to that in the other treatments (0.72 compared to 0.63–0.68, Table 5), as was also found by Gómez-Muñoz et al. (2016) However, it should further be noted that while the application of the treatments was performed by mixing with the soil, the sandy texture of the soil may have partly allowed NH_3 emissions to the atmosphere which acidification suppressed, contributing to higher plant yields in the acidified liquid treatment (Fangueiro et al., 2016).

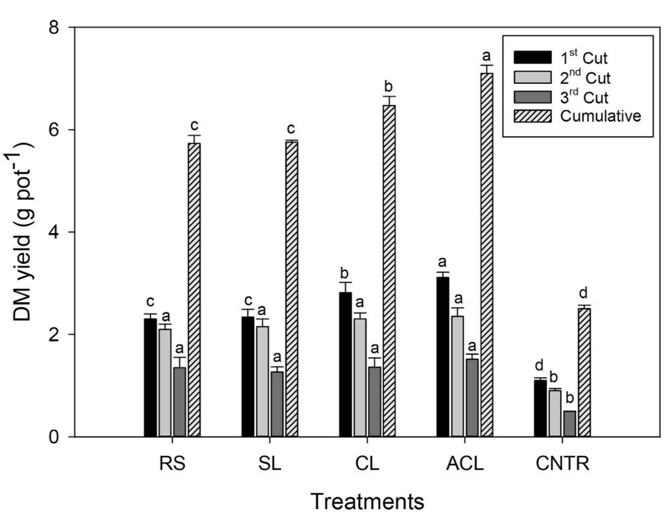

Fig. 2. Ryegrass dry matter (DM) yields (day 30, 60, 90 and cumulative) fertilized with raw slurry (RS), liquid fraction from screw press (SL), liquid fraction from centrifuge (CL) and acidified liquid from centrifuge (ACL). Error bars represent standard deviation of the mean (n = 4). Bars with different letters within each cut and cumulatively are significantly different ($p < 0.05$) according to Tukey test (HSD).

4. Operational costs and implications for the farm

The slurry treatment of the investigated farm aimed to produce an organic fertilizer that would allow crop fertilization to comply with both the Nitrates Directive suggesting maximum N application 170 kg N ha^{-1} $year^{-1}$ as well Helcom's recommendation to limit P application rates to 25 kg ha^{-1}. The separation system on the farm did not decrease the N fertilizing value of the liquid fractions when raw slurry and liquid effluents were applied based on their total N content in line with the Nitrates Directive. Being limited to the farm's operational conditions, the study lacks a direct comparison of the nutrient characteristics and the fertilizing value of the centrifuged liquid, without a preceding separation step. However, the examined slurry separation scheme, greatly reduced the amount of P introduced to the soil system with the different treatments, from 39.6 kg P ha^{-1} when unprocessed manure is applied to soil to only 11.8 kg P ha^{-1} when the separated and acidified liquid fraction was utilized as fertilizer. Close to 28 kg P may be reserved per hectare of soil by slurry separation. This would be important to reduce P surplus and P accumulation in the soil over time in order to decrease the risk of increased P losses to water bodies (Heckrath et al., 1995; Sims et al., 2000). For example, when calculating nutrient budgets for regions around the Baltic Sea, Svanbäck et al., (2019) found relationships between livestock density and nutrient surpluses, which indicated that redistribution of nutrients in manure to crop production areas would reduce nutrient load on the Baltic Sea in the long-term. Large-scale scenarios calculated by McCrackin et al. (2018) showed that redistribution of manure together with other agronomic measures have the potential to meet 38–64% of the reduction targets for P load on the Baltic Sea.

The current study provides further evidence of increased N utilization in acidified liquid slurries which possibly can compensate for the economic cost of acidification through reduced mineral fertilizers purchase. Sulfur requirements of arable crops range between 20 and 40 kg ha^{-1} (Pedersen et al., 1998) and the use of the non-acidified liquid slurry

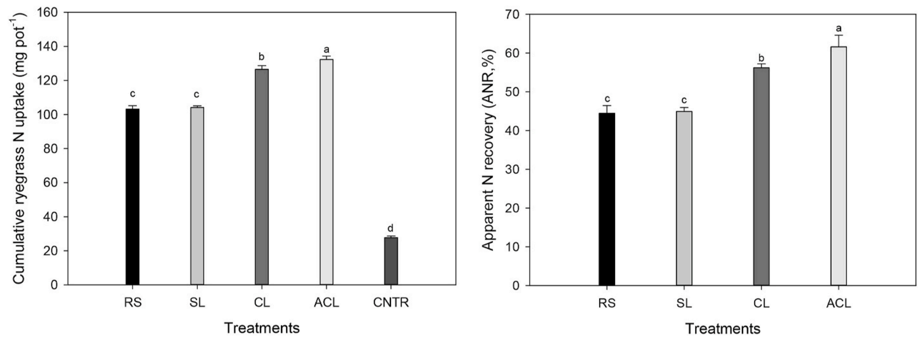

Fig. 3. Cumulative ryegrass yield N content (3 cuts) and apparent N recovery (ANR) from ryegrass fertilized with raw slurry (RS), liquid effluent from screw press (SL), liquid from centrifuge (CL) and acidified liquid from centrifuge (ACL). Error bars represent standard deviation of the mean (n = 4). Treatments with different letters are significantly different ($p < 0.05$) according to Tukey test (HSD).

of this study will add 17.8 kg S ha^{-1}, if the liquid is applied according to the limits set in the Nitrates Directive. Therefore, additional S fertilization might be needed for some high S demanding crops. In the current study, the acidified liquid fraction is expected to introduce to soil approximately 57 kg S ha^{-1} and exceed S crop demands. Long term use of acidified slurries may contribute to soil acidification and increase liming needs. However, use of acidifying slurries is not expected to substantially increase the soil's current liming needs derived from ammoniacal nitrogen nitrification and ammonia deposition. Nevertheless, the fertilization strategy should aim to balance the S application via the acidified slurries with plant uptake, to minimize potential S toxicity problems to plants and any risk associated with leaching of S especially in soils with low S absorption capacity.

Furthermore, storage of acidified liquid slurry may lead to a significant reduction in CH_4 emissions compared to bulk slurry storage (Supplementary material, S1). The potential of reduced climate impact from manure management poses a significant societal benefit which, should be taken into account from policymakers towards, promoting legislative and economic incentives for manure treatment implementation.

The solid fractions of the slurry represent a significant organic matter and P capital which can be further treated to facilitate their cost-efficient redistribution between regions. To that end, the double separation treatment of the slurry produces two diverse solid fractions with different possible utilization options. Solids from the screw press may be composted due to their high C content and light physical structure, while the P-rich solids from the centrifuge may be further treated to reduce their mass and volume to promote long distant transportation (Sommer et al., 2013).

The capital cost for implementing new technologies may be the most important challenge for a farm. However, higher nutrient utilization efficiencies and easier handling of manure may reduce costs associated with purchase of mineral fertilizers and equipment services.

5. Conclusions

In this study it was highlighted that a combined screw press and centrifuge separation scheme operated in full scale under real-farm conditions over a period of 5 months. Both efficiencies of equipment and mass balance of manure flows were investigated. It was found that such a system, with high load of manure, can provide high P separation efficiencies with the solid fractions over time, with preserved or increased N fertilizer value of the liquid fractions. Thereby this type of system may enable targeted application and reallocation of nutrients in slurries, which combine production and environmental aspects. The studied pig slurry management system can greatly balance the nutrient budget of the farm especially in cases where phosphorus is the excessive nutrient. The proposed treatment offers an attractive option for the rational utilization of nutrients from pig slurry especially in phosphorus vulnerable areas.

Furthermore, the two-step separation treatment of pig slurry enables total solids reduction and Cu and Zn removal from the liquid fractions. Subsequent acidification of solely the separated liquid fraction proves to be more cost efficient to acidification of the whole slurry and improve nitrogen availability for crops in sandy soil. The agronomic properties of the liquid effluents should be further studied in field trials with diverse soil characteristics.

The biochemical methane potential of the slurry is significantly reduced with a combined separation-acidification treatment and according to the estimations of this study may lead to lower CH_4 emissions during storage, which needs to be verified with on-site gas emission measurements.

CRediT author statement

Athanasios Pantelopoulos: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Writing - original draft, Writing - review & editing. Helena Aronsson: Conceptualization, Data curation, Formal analysis, Methodology, Writing - original draft, Writing - review & editing.

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.2020.111653.

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