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Effect of the pig slurry separation techniques on the characteristics and potential availability of N to plants in the resulting liquid and solid fractions

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Solid—liquid separation of slurry is used to obtain a solid (SF) and liquid fraction (LF), both used as organic fertilisers. Previous works showed that the separation technique used influences the composition of the resulting SF but no data are available relative to the LF. Four commonly used separation processes were considered — centrifugation, sieving, enhanced settling by the addition of cationic polyacrylamide (PAM-), and sediment settling as well as a recent sequential process — sieving followed by PAM addition to the resulting LF. The resulting LFs and SFs were comprehensively characterised and the amount of N potentially available for plants after incorporation to soil was also evaluated. Except for slurry that was only sieved which was very similar in composition to whole slurry (WS), the SFs and LFs had, respectively, higher and lower, concentrations of nutrients and organic matter (OM) than the WS. The ratio of ammoniacal to total N in the LFs varied from 0.33 to 0.92 whereas the values in the SFs varied between 0.16 and 0.31. The N:P:K ratio was also significantly influenced by the separation technique. Less than 10% of the total N applied in the WS, SFs or LFs was found to be potentially available for plants and, in some LFs, N immobilisation occurred after soil application. Since separation technique strongly influences nutrient distribution between LF and SF, as well as N speciation, the choice of the technique has to consider the final use of the resulting fractions as well as acquisition and operating costs.

1. Introduction

Pig production is often performed in specialised and intensive farms leading to the production of high volumes of liquid manure (slurry). Most of these specialised farms have no suitable fields to apply the slurry produced and this means they have to consider slurry treatment and/or export to other farms. Furthermore, even in non-specialised farms, slurry transport to fields is costly and energy consuming.

Solid—liquid separation of slurry is now widely used at farm level for slurry management (Hjorth, Christensen, Christensen, & Sommer, 2010). It leads to a solid fraction (SF) rich in phosphorous (P) and organic matter (OM), and to a liquid fraction (LF) which is rich in soluble nitrogen (N). The end products of slurry separation are generally applied to soil as source of nutrients and OM. According to Walker, Wade, and Kelley (2010), the soil application of SF and LF, rather than untreated slurry, reduces the risks of surface and

groundwater pollution and recent studies by Fangueiro, Trindade, Senbayram, and Chadwick (2008) showed that it can also reduce greenhouse gases emissions. Nevertheless, the SF can also be composted to provide a valuable commercial product, used for animal bedding (with or without thermal-treatment) or used as an energy source through biogas production or incineration (Menardo, Balsari, Dinuccio, & Gioelli, 2011). The LF can be used for fertigation or to flush the dairy house floor.

Several approaches have been considered to perform solid—liquid separation of pig slurry: active processes such as screening, sieving or centrifugation and passive processes, such as sedimentation or chemically enhanced sediment settling (Daumer, Picard, Saint-Cast, & Dabert, 2010; Hjorth et al., 2010). However, the use of sequential treatments' combining two or more separation processes, has been little investigated although previous laboratory experiments have shown that they can slightly increase the separation efficiency (Fangueiro, Pereira, et al., 2008; González-Fernández, Nieto-Diez, León-Cofreces, García-Encina, 2008).

Sneath, Shaw, and Williams (1988) showed that slurry centrifugation may reduce by about 15% the volume needed for storage of slurry and Asam et al. (2011) pointed out that it can also improve biomethane production by decreasing the thermal parasitic demand also in addition to decreasing transport costs and the size of facilities needed for storage.

Many studies dealing with slurry separation have focussed on the efficiency of separation techniques and given less importance to the quality/composition of the end-products (Møller, Hansen, & Sørensen, 2007; Møller, Jensen, Tobiasen, & Hansen, 2007; Møller, Lund, & Sommer, 2000). Jorgensen and Jensen (2009) showed that the physical and chemical characteristics of SFs depend on the technology used for separation but they did not investigate the LF.

Our hypothesis is that the distribution of nutrients between the SF and LF, as well as the N speciation in each fraction, depends on the separation technique used. The aim was to assess the chemical composition and fraction of plant available nitrogen of the SFs and LFs obtained from different separation techniques in order to distinguish between the available separation technique relative to different LF and SF uses.

2. Material and methods

2.1. Slurry separation

The pig slurry used in the experiments was sampled at a commercial pig livestock farm located in Rio Maior, Portugal. The slurry inside the store was mechanically stirred for ca. 30 min prior to sampling in order to obtain a homogeneous material. The slurry was stored at 4 °C in plastic barrels for two weeks prior the start of the separation process.

Five LFs (-L) and five SFs (-S) were obtained by: centrifugation at 1.509 × g for 15 min (Cent-), sieving through a 2 mm screen (Siev-), enhanced settling for 20 h by addition of 200 mg l^{-1} cationic polyacrylamide (VTA F 94) (PAM-), sediment settling during 20 h (Sed-), sieving followed by addition of PAM (for enhanced settling) to the resulting LF (Siev + PAM-). In the sedimentation and enhanced settling treatments, the LF was removed by pipetting the supernatant fraction. Slurry separation was performed on 1 kg of pig slurry and 4 replicates were considered for each separation process. The whole separation scheme used here is described in Fig. 1. These 10 slurry fractions as well as the whole slurry (WS) were comprehensively characterised as described below.

2.2. Analyses

The main characteristics of the WS, SFs and LFs were determined using standard methodologies described in Fangueiro et al. (2010). Briefly, total and mineral N concentrations were quantified by the Kjeldhal method and by extraction with 2 M KCl (1:10 w/v) followed by ammonium (NH_4^+–N) and nitrate (NO_3^-–N) quantification by molecular absorption spectrophotometry in a Skalar segmented flow analyser, respectively. Sampler of 100 g of fresh material were dried at 65 °C to constant weight for at least 24 h to quantify the dry matter content (DM). The OM was quantified by loss-on-ignition after incineration at 500–550 °C of
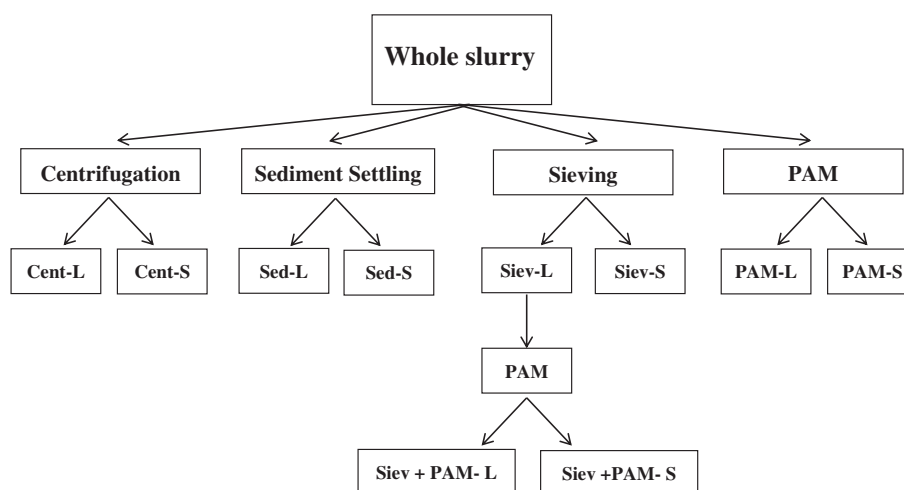
Fig. 1 – Slurry separation scheme.

the dry sample and total organic carbon (TOC) estimated from the OM value assuming that OM = TOC × 1.724 "Van Bemmelem Factor". Total potassium (K), calcium (Ca), P and magnesium (Mg) concentrations were quantified after wet ash digestion with hydrochloric acid in an atomic absorption spectrophotometer (PU 9000; PYE-Unicam Ltd, Cambridge, UK).

2.3. Potential plant available nitrogen

The procedure used here to assess the potential plant available nitrogen (PPAN) in the WS and the derived fractions was fully described in Fangueiro, Bol, and Chadwick (2008). Briefly, 10 g of field-moist soil were mixed with a specific amount of WS or slurry fraction equivalent to 30 mg N in a 60 ml syringe. The soil used was a sandy-loam soil. 8 replicates were constructed for each material and a treatment with soil only was run as control. Four were treated immediately to measure the initial NH_4^+–N concentration while the other 4 replicates were incubated for 7 d at 40 °C. The PPAN was calculated using the following equation:

$$PPAN = \left\{ \left(\left[NH_4^+\right]_{t=7} - \left[NH_4^+\right]_{t=0} \right)_{sample} \right.$$
$$\left. - \left(\left[NH_4^+\right]_{t=7} - \left[NH_4^+\right]_{t=0} \right)_{control} \right\}$$

where the PPAN was expressed as:

- % of the total N applied – PPAN (N_{tot}),
- % of the organic N applied when N mineralisation was observed – PPAN (N_{org})
- % of the inorganic N applied when N immobilisation was observed – PPAN (N_{inorg}).

The PPAN data were treated by analysis of variance (one way-ANOVA) and the least significant difference (LSD) test based on a t-test at a 0.05 probability level was used to assess the statistical significance of the mean differences.

3. Results and discussion

3.1. Main characteristics of the whole slurry, solid and liquid fractions

The proportion of LF and SF obtained varied significantly ($P < 0.05$) with the separation processes used (Table 1). The highest proportions of LF were obtained by centrifugation (67%) and sieving (55%) whereas all other processes led to 8–15% of LF. It should be noted that the quantification of the LF in passive processes is approximate and the values presented here are mainly indicative of the proportions obtained with the different processes. The proportions of LF and SF obtained here with the active processes were in the range of values reported by Hjorth et al. (2010).

The DM concentrations of the LFs obtained were significantly ($P < 0.05$) lower than from the WS and values were all very closed in the range 4.8–5.7 g kg^{-1} except the Siev-L treatment that had a significantly ($P < 0.05$) higher DM concentration (12.8 g kg^{-1}) (Table 1). If the purpose of slurry separation is mainly to remove solids in order to obtain a LF with low DM concentration, it may be concluded that the techniques used here were all suitable except that sieving was not so efficient. Pig slurry has generally a large fraction of small particle size (<0.1 mm) rich in DM (Fangueiro et al., 2010; Hjorth et al., 2010). Since a 2 mm sieve was used here, most of the slurry particles passed through the sieve and consequently only a small amount of DM remained in the SF. The DM concentrations of the SFs obtained were more variable with values from 80.0 to 251.7 g kg^{-1}, even if all in the range of values reported by Jorgensen and Jensen (2009). Such variability illustrates the capacity of each technique to concentrate solids even if the relative proportion of each fraction has also to be considered to accurately compare the separation efficiency (see Table 1). As occurred here, Hjorth et al. (2010) reported that the centrifugation is one of the most efficient techniques to concentrate the DM in the SF. If the SF has to be exported to other farms, it is profitable to have an SF with high DM concentration to avoid transport of water and the

Table 1 — Main characteristics of the whole slurry and slurry fractions obtained — mean and standard error of 4 replicates (wet weight basis).

	Proportion (% of WS)	DM (g kg⁻¹)	Total organic C (g kg⁻¹)	Total N (g kg⁻¹)	Total K (g kg⁻¹)	C:N$_{org}$ ratio	Total P (g kg⁻¹)	NH$_4^+$–N (g kg⁻¹)	NH$_4^+$–N: total N ratio	N:P:K ratio	Mg (mg kg⁻¹)	Ca (g kg⁻¹)
WS	/	82.5 (0.5)	21.4 (0.5)	4.2 (0.1)	1.8 (0.0)	7.4	1.1 (0.1)	1.3 (0.0)	0.3	1.0:0.2:0.4	350 (8)	3.7 (0.2)
Cent-L	67	4.9 (0.1)	1.1 (0.1)	1.4 (0.1)	1.1 (0.1)	4.8	0.02 (0.00)	1.2 (0.2)	0.8	1.0:0.02:0.8	49 (3)	0.2 (0.0)
Cent-S	33	251.7 (9.1)	68.5 (1.2)	10.4 (0.1)	1.7 (0.1)	7.8	5.7 (0.6)	1.6 (0.0)	0.2	1.0:0.5:0.2	927 (85)	11.7 (1.8)
Siev-L	55	12.8 (0.7)	4.4 (0.8)	4.1 (1.7)	1.4 (0.1)	1.6	0.04 (0.00)	1.3 (0.0)	0.3	1.0:0.01:0.3	101 (10)	0.5 (0.1)
Siev-S	45	160.1 (7.7)	52.9 (2.3)	6.0 (0.3)	1.5 (0.1)	11.3	3.5 (0.5)	1.4 (0.0)	0.2	1.0:0.6:0.2	474 (36)	8.4 (0.1)
Sed-L	12	5.7 (0.1)	1.3 (0.0)	1.4 (0.1)	1.0 (0.0)	11.4	0.04 (0.00)	1.3 (0.1)	0.9	1.0:0.03:0.7	70 (3)	0.2 (0.0)
Sed-S	88	83.5 (0.2)	23.8 (0.4)	4.3 (0.2)	1.3 (0.1)	8.0	1.8 (0.1)	1.3 (0.1)	0.3	1.0:0.4:0.3	333 (11)	3.7 (0.1)
PAM-L	8	4.8 (0.1)	1.1 (0.0)	1.4 (0.0)	1.0 (0.0)	3.6	0.04 (0.00)	1.1 (0.0)	0.8	1.00:0.03:0.73	50 (4)	0.1 (0.1)
PAM-S	92	80.0 (0.3)	22.9 (0.0)	5.0 (0.1)	1.3 (0.0)	5.9	1.9 (0.1)	1.2 (0.1)	0.2	1.0:0.4:0.3	301 (17)	3.4 (0.1)
Siev + PAM-L	15	4.9 (0.1)	1.1 (0.0)	1.4 (0.0)	1.1 (0.1)	3.7	0.04 (0.00)	1.1 (0.0)	0.8	1.00:0.03:0.82	51 (10)	0.29 (0.1)
Siev + PAM-S	85	88.1 (0.1)	22.6 (0.1)	4.6 (0. 1)	1.4 (0. 1)	6.8	2.3 (0.1)	1.3 (0.0)	0.3	1.00:0.50:0.31	394 (11)	3.7 (0.1)

consequent energy spent during active separation might be compensated for by the energy saved by transport relative to passive separation.

The organic carbon concentrations in the LFs and SFs obtained were significantly ($P < 0.05$) lower and higher than in WS, respectively (Table 1). As occurred with the DM concentration, similar values of organic C concentration were observed in the different LFs with a mean value of 1.1 g kg⁻¹ except in the Siev-L treatment where a higher value was observed. The high organic C content of the Cent-S and Siev-S fractions may be of interest if these materials will be applied to soil as a source of OM and, in such situation, the centrifugation and sieving separation techniques should be preferred.

In terms of total N, the SFs have similar concentration (4.3–6.0 g kg⁻¹) except the Cent-S fraction that has a significantly ($P < 0.05$) higher N concentration (Table 1). The LFs all had N concentrations in a range 1.4–1.4 g kg⁻¹ except the Siev-L fraction which had a value of 4.1 g kg⁻¹. The values of total N concentrations obtained here were within the range of values reported in previous works (Fangueiro et al., 2010; Fangueiro, Trindade, et al., 2008; Hjorth et al., 2010). The concentrations of NH$_4^+$–N varied from 1063.4 to 1626.7 mg kg⁻¹ in all the fractions obtained here and only small differences were observed between LF and SF. According to Jorgensen and Jensen (2009), even if the majority of mineral N remained in the LF, a significant amount of mineral N can also be found in the SF as some water is retained in the SF. In most fertilisation plans, the parameter to consider is the total N rather than the ammoniacal N even if in terms of plants nutrition, the NH$_4^+$–N supply is more important than the total N since only the NH$_4^+$–N can be directly used by plants. Hence it is important to consider the NH$_4^+$–N:N ratio when comparing organic fertilisers. The NH$_4^+$–N:N ratio of the SFs were all <0.3 whereas in the LFs they were all >0.7 except the Siev-L fraction (0.3). It might be expected that the LF application to grassland led to a stronger effect on plant growth than the SF, namely after a short time following soil application. Fangueiro, Trindade, et al. (2008) observed a higher increase of the grass yield following soil application of cattle LF than cattle SF.

The concentration of total K in the derived fractions varies between 1.0 and 1.7 g kg⁻¹ and the SF is generally richer in K than the LF for all the separation processes except sieving (Table 1). Inorganic K is very soluble (Hjorth et al., 2010) and should be found mostly in the LF whereas the organic K, being less soluble, should remain in the SF. The low K concentration found in LF may be due to the fact that the pig slurry used contained mostly organic K. In the case of the sieving process, the amount of SF retained in the screen was reduced due to the small particle size of the pig slurry and consequently the large amount of organic K found in the LF. Fangueiro, Ribeiro, Vasconcelos, Coutinho, and Cabral (2009) observed similar total K concentrations in LF and SF obtained by centrifugation of pig slurry indicating that the K separation efficiency depends on the initial WS composition.

The total P concentration of the LF is significantly ($P < 0.05$) lower than those of the respective SF with the higher differences observed in the case of the centrifugation and sieving processes. Moller, Sommer, and Ahring (2002) reported that

close to 7% of total P can be removed to SF using a screw press but Sommer, Maahn, Poulsen, Hjorth, and Sehested (2008) stressed that the P separation efficiency depends on the P concentration of WS that can vary strongly. The Cent-S and Siev-S samples have P concentrations higher than 3.5 g kg^{-1} which may be problematic. Indeed, considering the N:P:K ratio, these two fractions can be considered as strongly imbalanced and generally, the slurry application rates are defined to meet the crop N needs rather than crop P or K needs (Sharpley et al., 2003). As a consequence, an excess of P is usually applied to soil due to differences between the N:P ratio required by crops and the N:P ratio of the animal slurry (Leytem, Smith, Applegate, & Thecker, 2006). Nevertheless, the high P concentration from the Cent-S and Siev-S treatments can be considered as an advantage when soils are low in P since a single application of SF may ensure an efficient N and P fertilisation whereas the LF application should imply corrections using P mineral fertiliser. An application of excessive K via LF may be not so problematic since a large part is used by plants and this nutrient can accumulate in the soil and be used by a following crop. The difference between LF and SF in terms of N:P ratio may be used to improve the nutrients management at field scale since LF application to soil ensure a high available N application rate but a low P application rate whereas the opposite situation occurs when SF is applied to soil.

The Mg and Ca concentrations of the LFs obtained are significantly (P < 0.05) lower than those of the WS or any SF and varied from 49.5 to 101.7 mg [Mg] kg^{-1} and 127.6—488.6 mg [Ca] kg^{-1}, respectively (Table 1). The Sed-S, PAM-S and Siev + PAM-S fractions had similar concentrations of Mg and Ca but a significantly (P < 0.05) higher concentration of these elements were observed in the Cent-S. The separation technique used seems to have a poor effect on the distribution of these two elements between the LF and SF even if the active processes led to SFs with higher concentration in Mg and Ca relative to the fractions obtained with the passive process. Christensen, Hjorth, and Keiding (2009) reported that more than 80% of the Ca and Mg from pig slurry is in the particulate fraction and therefore, the active separation processes (namely centrifugation) was more efficient at separating DM and particulate material relative to passive processes and it will be more efficient at removing Mg and Ca from WS to SF.

3.2. Potential plant available nitrogen

The crop yield and quality rely on the quantity of N available for the plant. Hence, adequate amounts of N have to be supplied to fulfil crop requirements but excesses can lead to environmental problems due to N losses to air and water. A large part of slurry N is in organic form and its mineralisation following soil application depends on many parameters. The determination of the potential plant available N of slurry and slurry fractions is a useful tool to estimate the amount of organic N potentially available for plants and accurately define the amount of these materials to be applied.

N mineralisation was observed in the WS and all SFs considered here (Fig. 2) with values of PPAN in a range 2.7—6.3% of total N applied. This result was expected since all the SFs and WS have a C:N$_{org}$ value (see Table 1) lower than 15 and according to Chadwick, John, Pain, Chambers, and Williams (2000), such material should lead to N mineralisation. Nevertheless, the same authors stressed that N mineralisation/immobilisation cannot be predict considering only one parameter. The evolution of N (mineralisation or immobilisation) in the LFs depended on the separation technique: N mineralisation was observed in Sed-L and PAM-L whereas N immobilisation was observed in Siev + PAM-L and Siev-L; the Cent-L fraction did not lead to either N mineralisation or immobilisation. It be noted that the higher and lower values of PPAN were observed in PAM-L and Siev + PAM-L, respectively. No significant (>0.05) relationship was found between N mineralisation and any chemical characteristics of the LFs even if the C:N organic ratio, the NH$_4^+$—N:N$_{tot}$ ratio, lignin:N ratio have been pointed out as good indicators of the N availability for plants (Fangueiro et al., 2010; Fangueiro, Pereira, et al., 2008). It was shown that the particle size of slurry has also a significant effect on the PPAN (Fangueiro et al., 2010) and it is to believe that the separation processes used here will lead to LFs with distinct particle size fractions which may explain the differences observed here. More than 20% of the mineral N was immobilised in the Siev + PAM-L fraction and more than 20% of organic N was mineralised in PAM-L treatment (Fig. 3). In all other treatments with positive PPAN values, ∼5% of organic N was mineralised. The amounts of organic N potentially available for plants observed here were similar to those reported in previous works (Fangueiro, Bol, et al., 2008; Fangueiro et al., 2010). The small PPAN
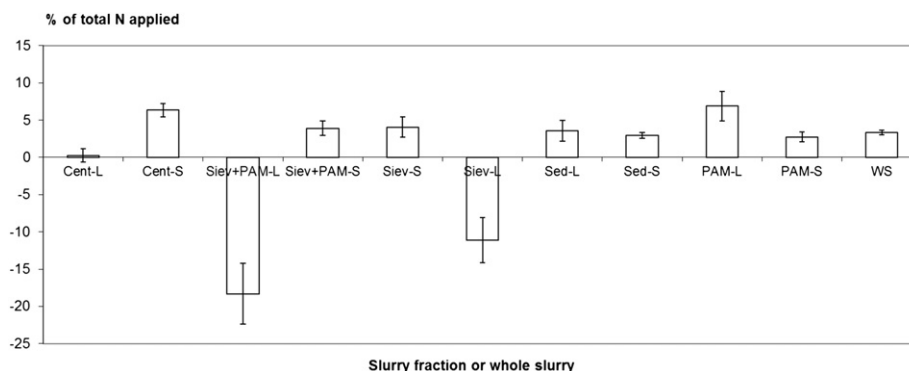
Fig. 2 — Potential plant available N from the whole slurry and slurry fractions expressed as % of total N applied.
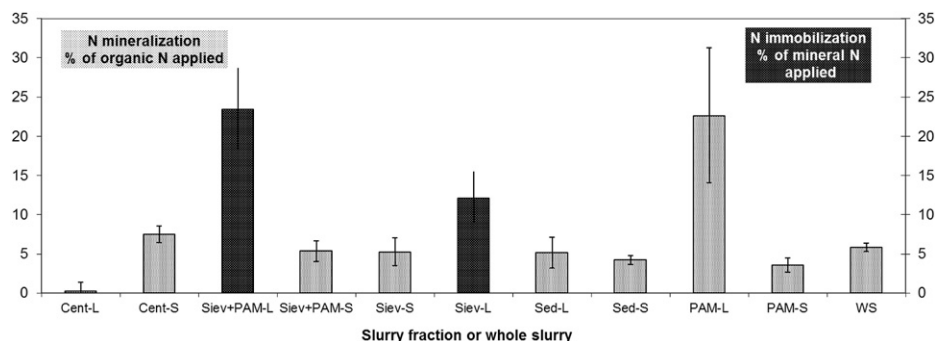
Fig. 3 – Potential N mineralisation/immobilisation from the whole slurry and slurry fractions expressed as % of organic N or mineral applied. N mineralisation was observed in treatments shown with grey columns and N immobilisation was observed in treatments quoted with black columns.

values in some fractions may indicate that the organic N in these fractions is strongly bound and less prone to rapid release. However, negative PPAN values may be interpreted as indication of a large amount of readily available N that can be used by plants immediately after application or even used by soil microorganisms when not removed by plants. Hence, it might be of interest to apply fractions with positive and higher PPAN values before sowing (before plants uptake) to allow N mineralisation to occur. Fractions with negative PPAN values could be used for the production of crops with high demands of mineral N in short time intervals and should be applied preferentially during crop growth rather than before sowing.

3.3. Implications of slurry separation at farm scale

Our results show that both passive and active separation techniques (except sieving) efficiently remove most solids from the LF enabling an easier application to the soil either by injection or by umbilical systems. In some cases, slurry treatment has been assumed as a solution allowing direct discharge of LF to water bodies but Vanotti and Hunt (1999) considered that most of the equipment available for slurry separation was not efficient for total nutrient removal (i.e. N, P, K) from LF to SF and consequently, LF obtained by mechanical separation should not be directly discharge to water bodies without further treatment. Indeed, Fangueiro et al. (2010) recently showed that even screening to 100 μm was not enough to remove significant amounts of N and P. Therefore, the fractions obtained by separation should preferably be applied to soil as organic fertiliser. Our data showed that the composition of the resulting fractions depends on the separation technique and thus the choice of the separation technique has to consider the final use of the resulting fraction. Nevertheless, one has to keep in mind that technologies to perform active separation are expensive and energy consuming whereas the passive separation implies limited investment.

The environmental impact of slurry separation has also to be considered since previous studies show that slurry separation may in some way extend, increase or reduce greenhouse gases emissions, as well as nutrient leaching to water bodies, following soil application (Chadwick et al., 2011; Pietro, Pruneddu, Giunta, Jones, & James, 2012). Indeed, the LF is

richer in NH_4^+–N than SF so it should lead to higher ammonia (NH_3) emissions than WS. However, an LF with a low DM content should infiltrate in soil more rapidly than an LF with higher DM content so NH_3 emissions should be short term and it may also affect carbon dioxide (CO_2) and nitrous oxide emissions (Bhandral et al., 2009). On the other hand, this same DM-low LF will reach deeper soil layers and may consequently lead to higher nutrient losses by leaching causing water pollution (Smith, Beckwith, Chalmers, & Jackson, 2002). SF rich in DM should also lead to higher CO_2 emissions even if most of C in SF is more recalcitrant (poor mineralisation) whereas in LF, most C is labile and easily lost as CO_2 (Fangueiro, Chadwick, Dixon, & Bol, 2007).

Separation processes using polymers, such as PAM, are of interest since they may increase the separation efficiency of some elements, namely P (Hjorth, Christensen, & Christensen, 2008) but their use also implies a significant increase in slurry treatment costs. Nevertheless, Walker et al. (2010) reported that a "static gravity screen-roll press separator operated in tandem with a PAM assisted gravity belt thickener", similar to our Siev + PAM process is economically viable when the LF is used for fertigation. Polymer use should therefore be considered as an integrated solution for slurry management at farm scale (including SF and LF utilisation) and not only as a slurry treatment option otherwise it may not be economically sustainable.

Our results show that centrifugation is the most efficient process to separate DM and nutrients. Furthermore, it leads to two contrasting fractions: a SF with the highest DM and total nutrients contents but low NH_4^+–N:total N ratio; a LF with low DM and total nutrients contents but high NH_4^+–N:total N ratio and low PPAN. The plant requirements in term of nutrients vary widely and good timing for application to the soil may help to prevent losses. To achieve this mineral N will have to be applied during plant growth to ensure that most is used by plants and not lost by leaching whereas the organic N has to be applied before sowing in order to allow N mineralisation to occur in the soil prior to plant removal. Consequently, the Cent-SF fraction, with low mineral N concentrations and high concentrations of total P and N, should be applied before sowing whereas the Cent-LF, with higher mineral N concentrations, should be applied during plant growth.

4. Conclusions

The active and passive separation processes investigated led to significant differences in the distribution of nutrients between the resulting LF and SF. Centrifugation appears as the most efficient to separate DM and nutrients and this led to a significant amount of LF. The passive processes have a lower separation capacity and generated only 10–15% of LF even if the DM and nutrient separation were similar to those obtained with active processes.

All the LFs obtained have high NH_4^+–N:total N ratios and, consequently, were more suitable for soil application during plant growth whereas SFs should ideally be applied before sowing to allow for mineralisation of organic N.

According to our results, the choice of the separation technique has to consider, not only the acquisition and operation costs or DM separation efficiency, but also the final application of the end-products and consequently the required characteristics of the resulting fractions.

Acknowledgements

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