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Anaerobic Digestion and Microfiltration of the Liquid Fraction of Pig Slurry: N Mineralization, C-CO$_2$ Emissions and Agricultural Value of the Products

Miguel Fernández-Labrada[1] · María Elvira López-Mosquera[2] · Adolfo López-Fabal[1]

Abstract

Pig slurry contains valuable nutrients and organic matter, although its high water content makes its management and use as a fertilizer more expensive. It is also an interesting bioenergy resource for biogas production. We propose a treatment that consists of solid–liquid separation followed by the anaerobic digestion of liquid fraction of slurry (LFS) while a microfiltration membrane module concentrates the solids in the digester and removes a liquid fraction of the digestate (LFD). The aims of the work were to evaluate the fertilizer value of the LFS, digestate and concentrated digestate and the possibility of reusing the LFD in agriculture. The LFS contained 72% less dry matter than the slurry. The solid–liquid separation mainly removed N and P. Thanks to microfiltration, the remaining solids were partially recirculated to the digester, concentrating there. To do this, the membrane module continuously removed the LFD, which was made up of 99% water with dissolved elements, mainly C, N, K and Na. The concentrated digestate contained less K, similar amounts of N and P, and more dry matter and C than the initial LFS. The entire slurry treatment affected the mineralization dynamics by increasing recalcitrant C and decreasing labile C without modifying N release. The proposed process allowed taking advantage of the nutrients and stabilized organic matter contained in the LFS, producing a concentrated digestate. LFD did not meet the reclaimed water requirements. However, it could be useful as a fertirrigation solution and a post treatment could be enough to comply with the regulations.

Graphical Abstract
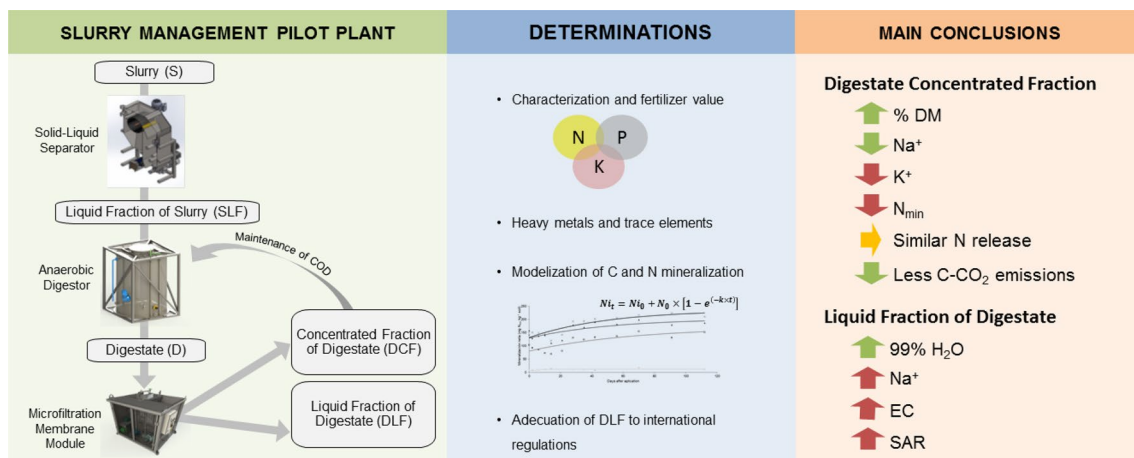
Keywords Modelling · Reclaimed water · Solid–liquid separation · Digestate · Labile carbon · Recalcitrant carbon

✉ Adolfo López-Fabal
adolfo.lopez@usc.es

Extended author information available on the last page of the article

Statement of Novelty

A new pilot plant to digest the liquid fraction of slurry was studied. Microfiltration was used to remove the water from the digester and recirculate the solids. The main novelty of this study lies in the processing of the slurry. While solid-liquid separation is widely studied, the proposed treatment of the liquid fraction is unusual. This article studies extensively how this new treatment affects the traditional use of slurry as a fertilizer. The amount, balance and nutrient release rate of the produced fractions were measured. Furthermore, it is evaluated how the process could affect the C stock in the soil. Following the circular economy production model, the possibility of using the liquid fraction of the digestate as reclaimed water is considered.

Introduction

Pork meat is largely produced on intensive farms which generate large amounts of pig slurry that is usually reused as an organic fertilizer [1, 2] on the grounds of its high contents in nutrients and organic matter. For easier handling, and more economical transport and use, the slurry is often split into a solid phase and a liquid phase [3] that differ in agronomical value. Thus, the solid fraction contains most of the carbon and nutrients —in organic form—, and hence little moisture [4], which facilitates inexpensive transfer of nutrients from slurry-rich areas to nutrient-deficient areas [5]. On the other hand, the liquid fraction consists mainly of water but additionally contains substantial amounts of dissolved elements such as N and K. These characteristics improve the efficiency of the nutrients since they are provided directly in forms that can be assimilated by the crops and, furthermore, by rapidly infiltrating the soil, N losses to the atmosphere are reduced [6, 7]. Because of its high moisture content, however, the liquid fraction of slurry is expensive to transport and apply, which somehow restricts its use as a fertilizer [8].

The liquid fraction of slurry can be valorized by anaerobic digestion to obtain biogas and a by-product (digestate) with distinct agronomic properties. Anaerobic digestion converts the most labile fractions of organic matter to CO_2 and CH_4, thereby increasing the biological stability of the digestate and reducing its contents in total C and dry matter. During the process, N is partially mineralized and accumulates as $N-NH_4^+$, which decreases the C/N ratio [9–12]. By using only the liquid fraction, hydraulic retention time is reduced, C removal is improved, and there are fewer operational problems [13–16]. Anaerobic digestion provides additional advantages such as reducing

off-odours [17], pathogens [11] and greenhouse gas emissions [18]; however, the resulting digestate still consists mainly of water.

One efficient way of concentrating nutrients present in slurry digestates is by filtration across membranes and microfiltration is the most recommended given that retain most of the nutrients, are scarcely prone to clogging, have a high throughput and are operationally inexpensive [19, 20]. In fact, microfiltration can reduce biochemical oxygen demand (BOD) by 76–89%, total N by 12–38.5%, total P by 44.6–75% and total solids by up to 37% [20, 21]. The treatment provides a concentrated effluent containing the nutrients in addition to waste water that can be used for irrigation, industrial use (refrigeration, dust control) or urban maintenance purposes (garden irrigation, street hosing) [22].

The recirculation of the solid fraction of the digestate has been tested as a method to improve the efficiency of the anaerobic digestion process [23, 24]. However, in no case that we know of, the implications this process may have in the subsequent reuse of the digestate as fertilizer are studied. Both the separation process and the anaerobic digestion treatment alter the total amounts of C and N, their ratio and their chemical distribution, thereby potentially modifying their dynamics in soil and their fertilizing capacity [25–27]. In fact, the soil dynamics of C and N is influenced by the contents of the two elements and their ratio. Thus, supplying soil with large amounts of labile, easily degraded C causes an exponential increase in microbial activity. Also, the C/N ratio dictates whether N will be preferentially mineralized or immobilized during microbial growth [7].

The primary aim of this work was to identify the changes in composition and properties governing the potential of the liquid fraction of pig slurry, and the elements of its anaerobic digestion and microfiltration, as fertilizers. Because both treatments were expected to alter the composition and C/N ratio, we also examined and modelled the mineralization dynamics of the two elements in soil. In addition, were characterized the liquid fraction of the digestate to assess its potential use as recycled water (specifically, irrigation water).

Material and Methods

Slurry Processing Plant

It was used an anaerobic digestion pilot plant located in Cospeito, Lugo (Galicia, north-eastern Spain). In this work, the plant was exclusively used to process the slurry liquid fraction, which was concentrated by using a membrane module during and after digestion. The plant comprised a slurry tank, a rotating-sieve solid–liquid separator of 1 mm mesh (TAGA ATR) and a 4.5 m³ anaerobic digester equipped

with an helical stirrer (TIMSA HRT-03 17 L01) and heating wires to maintain a constant temperature. The digester was followed by a microfiltration module of synthetic polymer membranes (0.1 μm pore size, 6.25 m² surface area, − 15 to − 35 kPa transmembrane pressure) (TAGA MBR07). Microfiltration was carried out in sequences of 10 min of work and 3 min of rest. During rest, a microbubble diffuser system used the biogas to create an air current that dragged the layers that cover the membranes. This allowed the same membrane to be used throughout the process. After the module of microfiltration, two tanks were placed to collect the resulting fractions.

Slurry Treatment and Fractions Obtained

The slurry (S) was obtained from a conventional pig fattening farm in the vicinity of the plant (the main characteristics are described in Table 2). As required it was subjected to solid–liquid separation, the resulting liquid fraction (SLF) being fed to an anaerobic digester at a rate of 0.15–0.40 m³ day⁻¹. The digester was operated in the mesophilic range (37 °C), using a hydraulic retention time of 10–20 days. On an intermittent basis, a portion of digestate (D) in the digester was fed to the membrane module for microfiltration several times a day. This operation provided a liquid fraction (DLF) that was stored in a tank and a concentrated fraction (DCF) that was recycled to the digester in order to ensure a continuous organic load, as chemical oxygen demand, of 2–4 kg·m⁻³. If the organic load was adequate, the DCF was sent to a storage tank.

Characterization of Fractions

With the plant operating under its usual regime, samples of the different fractions were obtained over a period of 4 weeks. By exception, only a single sample of slurry (S) was taken from the storage tank since it was homogeneous and not changed throughout the sampling period. SLF, D and DCF were sampled on a weekly basis through stopcocks inserted in the processing line. Unlike the previous fractions, DLF was sampled twice a week at the point of discharge. Samples were stored refrigerated at 4 °C until analysis.

DLF Analysis

Fresh samples were analysed for (*a*) pH and electrical conductivity; (*b*) turbidity with a Dr. LANGE LTP4 turbidimeter; (*c*) NO_3^- and NH_4^+ with ion-selective electrodes; (*d*) suspended solids by vacuum filtration through a micro-fibreglass filter of 0.45 μm pore size; (*e*) total solids by evaporation at 105 °C; (*f*) C and N simultaneously on a LECO TruSpec combustion analyser; and (*g*) P, K, Ca, Mg, Na, Cd, Cu, Cr, Hg, Ni, Pb, Zn, As, B, Co, Mn, Mo, Se and

Table 1 Amounts of fresh and dry matter, water, N and C supplied by each product to the soil in the incubation tests

Component	S	SLF	D	DCF
FM (g kg⁻¹ soil)	46.64	111.08	96.87	117.16
DM (g kg⁻¹ soil)	2.85	0.13	2.13	6.20
H₂O (mL kg⁻¹ soil)	67.17	0.00	16.22	0.00
Mineral N (mg kg⁻¹ soil) *	60.15	2.53	69.68	59.66
Total N (mg kg⁻¹ soil)	156.99	4.13	107.61	229.32
C (mg kg⁻¹ soil)	1455.67	41.84	798.21	2557.40

S slurry; *SLF* slurry liquid fraction; *D* digestate; *DCF* digestate concentrated fraction; *FM* fresh mater; *DM* dry matter

*Sum of N–NH_4^+ and N–NO_3^-

V by inductively coupled plasma mass spectrometry (ICP-MS). The last results were used to calculate the Sodium Adsorption Ratio (SAR) acording Eq. 1, where [Na^+], [Ca^{2+}] and [Mg^{2+}] are the concentration of the respective ions (mmol(+) L⁻¹)

$$SAR = \frac{[Na^+]}{\sqrt{\frac{[Ca^{2+}]+[Mg^{2+}]}{2}}} \tag{1}$$

S, SLF, D and DCF analysis

Fresh samples were used to measure pH, electrical conductivity and dry matter. An aliquot each of S, SLF, D and DCF that was previously frozen and freeze-dried was used to determine C and N on the LECO autoanalyser; N–NO_3^- and N-NH_4^+ with ion-selective electrodes in a 1:25 (w/w) resuspension after hydration with ethanol; and P, K, Ca, Mg, Na, Cd, Cu, Cr, Hg, Ni, Pb and Zn by ICP-MS following microwave-assisted digestion in nitric acid [28].

Incubation Tests

The mineralization dynamics of C and N were studied for S, SLF, D and DCF upon their application to soil. A laboratory test was performed using the surface layer (0–20 cm) of an extensive cropland in the vicinity of the pilot plant. Samples were air-dried and sieved through 2 mm mesh prior to incubation. The soil was sandy loam in texture, had pH 6.34 in a 1:2.5 water suspension, and an electrical conductivity of 0.121 dS·m⁻¹ in a 1:5 suspension; also, it contained 5.40% organic matter and 0.247% total N (dry combustion); 21.3 mg N–NO_3^- kg⁻¹ and 16.8 mg N–NH_4^+ kg⁻¹ (as described below) and 32.6 mg Olsen P kg⁻¹. The cation contents extracted by 1 N NH_4Cl and analyzed in ICP-AES were 7.65 (Ca^{2+}), 1.53 (Mg^{2+}), 0.13 (Na^+), 1.09 (K^+) and 0.05 (Al^{3+}), all in cmol(+) kg⁻¹.

Table 2 General properties of the starting slurry and the fractions obtained from its processing

Property	S	SLF				D				DCF			
		Mean		SD		Mean		SD		Mean		SD	
pH	7.04	7.84	±	0.51	b	7.75	±	0.25	ab	7.61	±	0.17	a
EC (dS m^{-1})	18.54	8.24	±	0.87	a	12.95	±	0.51	c	11.85	±	0.63	b
DM (g kg^{-1})	35.55	7.86	±	1.43	a	19.90	±	4.36	b	56.92	±	9.43	c
C (g·kg^{-1} d.m.)	321.52	331.43	±	109.23	ab	378.11	±	6.32	a	410.93	±	3.78	b
N (g·kg^{-1} d.m.)	85.28	41.62	±	7.99	a	43.82	±	9.30	a	36.25	±	4.44	a
C/N	3.77	7.96	±	1.67	a	8.63	±	1.94	a	11.34	±	1.58	a
N–NO$_3^-$ (g kg^{-1} d.m.)	n.d	16.26	±	5.35	b	14.31	±	14.31	b	4.82	±	1.90	a
N–NH$_4^+$ (g kg^{-1} d.m.)	n.d	18.93	±	4.86	b	13.14	±	13.14	b	6.14	±	0.71	a
P (g kg^{-1} d.m.)	8.56	6.56	±	2.77	ab	7.51	±	0.32	a	8.66	±	0.50	b
K (g kg^{-1} d.m.)	127.27	110.52	±	13.11	c	60.48	±	5.21	b	26.58	±	4.11	a
Ca (g kg^{-1} d.m.)	36.85	24.69	±	10.89	a	30.01	±	1.32	a	27.71	±	1.96	a
Mg (g kg^{-1} d.m.)	8.46	7.34	±	2.62	ab	8.83	±	0.32	b	7.82	±	0.30	a
Na (g kg^{-1} d.m.)	33.15	28.83	±	3.10	c	13.75	±	1.28	b	5.96	±	1.17	a

S, slurry; *SLF*, slurry liquid fraction; *D*, digestate; *DCF*, digestate concentrated fraction; *FM*, fresh mater; *DM*, dry matter

*Sum of N-NH$_4^+$ and N-NO$_3^-$

In order to avoid an initial increase in microbial activity by effect of the favourable temperature and moisture conditions, the soil was previously moistened and incubated in the absence of fertilizer for 2 weeks.

Nitrogen mineralization was quantified by incubating an amount equivalent to 600 g of dry soil with the proportional dose of fertilizers (Table 1) in 1 L hermetic plastic boxes, which were aerated at least once a week. Periodically, samples of 25 g of fresh soil were taken and supplied with 75 mL of 2 M KCl and passed through Whatman no. 42 filter paper after stirring for 1 h. The resulting extract was analysed for ammonium ion with the Indophenol Blue colorimetric method [29], and also for nitrate ion by colorimetric measurement after reduction to nitrite ion with hydrazine sulphate [30].

Carbon mineralization was evaluated by incubating an amount equivalent to 50 g of dry soil with the proportional dose of fertilizer (Table 1). The soil was placed in tight-closed containers also holding a vial containing an alkaline solution (1 N NaOH) to trap CO_2 and another containing water to avoid desiccation. At each sampling time, the NaOH vial was titrated with 0.5 N HCl following precipitation of carbonate ion with $BaCl_2$ [31] and replaced with another containing fresh NaOH solution.

In both incubations the same dose of each fertilizer was added to the soil. An amount of S, D, and DCF equivalent to approximately 60 mg mineral N·kg^{-1} dry soil (Table 1) was applied, adding water later to achieve 60% field capacity. No similar amount of SLF could be used because this product had a very high moisture content that limited the total amount of fertilizer it supplied. Four replicates were performed for each treatment and the control (no fertilizer), so each test had 20 experimental units.

Both incubation tests were conducted simultaneously at 25 °C in the same chamber for 16 weeks. Samplings were done simultaneously at a variable frequency on the days 2, 6, 10, 14, 21, 28, 35, 42, 56, 70, 91 and 112.

Modelling of Mineralization Curves

The results of the C and N mineralization tests were modelled in two different ways:

(a) With the single-fraction first-order kinetic model of Stanford and Smith (1972), based on Eqs. 2 and 3:

$$Ni_t = Ni_0 + N_0 \times \left[1 - e^{(-k \times t)}\right] \quad (2)$$

$$Ci_t = C_0 \times \left[1 - e^{(-k \times t)}\right] \quad (3)$$

where Ni_t is the amount of soil inorganic nitrogen present at time t, Ni_0 the initial amount of inorganic N, N_0 that of potentially mineralizable N, k the N mineralization rate constant (day^{-1}) and t time (days) in eq. 2; and Ci_t is the amount of C released at time t, C_0 that of potentially mineralizable C, k the C mineralization rate constant (day^{-1}) and t time (days) in eq. 3.

(b) With the two-fraction first-order kinetic model of Molina et al. (1980), based on Eqs. 4 and 5:

$$Ni_t = Ni_0 + N_1 \times \left[1-e^{(-k1\times t)}\right] + N_2 \times \left[1-e^{(-k2\times t)}\right] \tag{4}$$

$$Ci_t = C_1 \times \left[1-e^{(-k1\times t)}\right] + C_2 \times \left[1-e^{(-k2\times t)}\right] \tag{5}$$

where Ni_t is the amount of soil inorganic nitrogen at time t; Ni_0 is the initial amount of inorganic N; N_1 and N_2 are the amounts of the two fractions of potentially mineralizable N; k_1 and k_2 the mineralization rate constant for N_1 and N_2, respectively (days^{-1}); and t is time (days) in eq. 4; and Ci_t is the amount of C released at time t; C_1 and C_2 are the amounts of potentially mineralizable C in the two fractions; k_1 and k_2 the respective mineralization rate constants (day^{-1}); and t is time (days) in eq. 5.

The modelled results were used to estimate mineralizable N (Ni_t) and the fractions of carbon (mineralized and recalcitrant).

Statistical Analysis

The results were analysed statistically by using the software SPSS Statistics v. 25 from IBM Corp. (Armonk, NY, USA). Data were checked for normality with the Shapiro–Wilk test, failure of which led us to adjust them to a normal distribution by appropriate transformation. Variances were checked for homoscedasticity by using Levene's test. The significant treatments were compared through Tukey's HSD multiple range test ($p < 0.05$) or T3 Dunnett's test ($p < 0.05$) if the variables did not fulfil the homoscedasticity criterion.

The effect of each treatment was assessed from the variation rate of the concentration of each element and dry matter as follows:

$$Variation\ rate = \frac{concentration\ after\ (w/w) - concentration\ before(w/w)}{concentration\ before\ (w/w)}$$

The dynamics of C and N mineralization were modelled with the specific choice best fitting the experimental results. Significant differences between curves were identified by processing the results of each treatment individually (whole model) and all in combination or in pairs (reduced models). The whole model was compared with the reduced models via an F-test:

$$F = \frac{\left(SSE_r - SSE_f\right)/\left(df_r - df_f\right)}{\left(SSE_r - df_f\right)}$$

where SSE_r and SSE_f are the sums of squares of the reduced and whole model, respectively, and df_r and df_f are the corresponding degrees of freedom. A reduced model was deemed inapplicable, and differences between treatments

were assumed to be significant as a result, when its difference from the whole model led to $p < 0.001$.

Results and Discussion

Characterization of Fractions

Slurry Liquid Fraction

The rotating sieve used to separate the SLF of the slurry retained particles larger than 1 mm, thereby reducing the dry matter (DM) content of the slurry by 78% (Table 2). This separation efficiency is greater than previously reported values [34], especially if one considers that the starting slurry had a relatively low DM content [35]. Also, the separation efficiency of slurry components is known to be widely variable (11–87%) [3]. Solid–liquid separation has proved an effective choice for enriching the solid fraction with most of the dry matter, N and P present in pig slurry [36, 37]. In this work, the N content of the SLF was reduced by no less than 51%; also, because the C content was scarcely altered, the C/N ratio was nearly doubled as a result. This was a consequence of a substantial proportion of total N (and, especially, organic N) in pig slurry being present in large particles [25] —in fact, 85% of all N in SLF was inorganic. Although the results suggested a reduction in P content by effect of solid fraction of slurry being removed, no clear-cut conclusion can be drawn in this respect because the data were rather variable. On the other hand, the K, Ca, Mg and Na contents differed little between the slurry (S) and its liquid fraction SLF, which suggests that these elements remained largely in the former —something that was to be expected since the previous elements are mostly present in soluble forms bound to particles 0.45–50 μm in size [38].

Digestate

Through digestion, a fraction of organic matter is mineralized to simpler compounds, carbon dioxide and methane [39]. This usually increases the proportion of soluble nutrients, and decreases that of carbon and dry matter, in the resulting digestate [40]. In this work, however, we observed the opposite trend. Thus, digestion decreased N–NO$_3^-$, N–NH$_4^+$, K and Na levels by 12, 31, 45 and 52%, respectively, while DM increased by 153% (Table 2). This was the likely result of the digestate being continuously filtered

through the membrane module and the concentrated fraction being recycled while the liquid fraction DLF was removed together with dissolved salts (i.e., of a washing effect). On the other hand, the Ca and Mg levels were increased by about 20%. These elements were not in soluble form but rather bound to particles 0.45–50 μm in size [38], which prevented them from crossing the membranes and, as a result, remaining in the DCF and being returned to the digester in concentrated form.

Carbon levels should also have been reduced by effect of the formation of CH_4 and CO_2 [41]. However, recycling caused all C bound to the larger particles to be returned to the digester while other soluble elements were washed off, C levels increasing by 14% as a result. Thus, despite the low OM content of the SLF, the digester maintained an adequate organic load by effect of excess water and soluble salts being removed, and the residence time of lignocellulosic materials —which are slower to hydrolyse— being expanded [42].

Digestate Concentrated Fraction

Microfiltration of the digestate led to most dry matter in it passing into the DCF, which in fact contained an amount of solids 2.86 times greater than that of the D itself and 7.24 times more so than the SLF (Table 2). Microfiltration also increased the content in P (by up to 15%), which was present mostly in the solid fraction [36].

As stated above, microfiltration removed salts that were transferred to the DLF. This reflected in a decrease in electrical conductivity in the DCF relative to the D itself. The salts exhibiting decreased levels by effect of microfiltration included those of N. Thus, nitrate and ammonium ion levels were reduced by 66 and 53%, respectively. This led to a slight decrease in total N which, however, was not significant, and especially, to the inorganic fraction of N being more than halved (from 63 to 30%) —which had strong implications on its potential as a fertilizer. There was also a decrease in the contents of K and Na, which were sent largely to DLF as a result of their being mostly (more than 90%) in dissolved form or bound to particles less than 0.45 μm in size [38].

Waeger et al. (2010) previously reported a reduction of 60% (w/w) in ammonium ion levels in addition to an increase in C and P levels (69 and 60%, respectively) by effect of slurry digestate being concentrated through microfiltration. Chiumenti et al. [43] obtained similar results by passing the liquid fraction of a digestate through a membrane of 0.1 μm pore size; the levels of soluble ions such as K^+ and $N–NH_4^+$ were decreased by 19 and 13% (w/w), respectively, whereas P was largely retained in the solid fraction, with an increase by 68%. The more marked reductions in soluble ions (K^+ and $N–NH_4^+$) and reduced P retention capacity observed here may have resulted from recycling allowing

most elements retained in organic or exchangeable forms being converted into soluble forms. Heavy recycling of DCF was required in order to maintain an adequate organic load in the digester —one higher than that of SLF, with which it was fed— but would be unnecessary or much less of a requirement if the whole slurry (S) were treated. Therefore, through the combined digestion-microfiltration process, the main disadvantage of digesting the liquid fraction is overcome; a low organic load that implies less biogas production per volume unit [14]. In addition, the main advantages of using only the liquid fraction are maintained; shorter hydraulic retention time [14], higher biogas production per unit of dry matter [13, 14], fewer pumping, mixing or clogging problems [15, 16].

The elements bound to the larger particles (Ca, Mg) should have been returned to the digester and their concentrations in DCF been very similar to those in the influent (D). This was in fact the case with Ca, which remained at steady levels throughout, but not with Mg, whose levels were reduced by up to 10%.

Although the solid fraction of slurry was removed at the start, the properties of the resulting DCF showed similarities to those of the starting slurry (S). Such was the case with the Ca, Mg and P contents, but not with the dry matter and C contents, which were 60 and 28% higher, respectively, in DCF. On the other hand, the levels of N and K were reduced by 57 and 80%, respectively, as a result of their washing to DLF. These changes increased the C/N ratio from 3.77 in S to 11.34 in DCF and must have influenced the soil dynamics of C and N [44, 45]. Nutrient balances of N/K were also altered. Thus, the N/K ratio, which is known to influence fertilizer performance [46], increased from 0.67 in S to 1.36 in DCF. Therefore, the N:K ratio would not be as suitable for some K-demanding crops such as potatoes (N:K ratio 0.6) [47] or sugarcane (N:K ratio 0.5) [48]. However, it could be used in N-demanding crops such as corn (N:K ratio 1) [49] or ryegrass (N:K ratio 0.8) [50] and in top-dressing where it is sought to provide mainly nitrogen.

Digestate Liquid Fraction

Microfiltration proved effective to remove moisture from the D, which was 99% water (Table 3). This was also the case with suspended solids, which accounted for only 3% of all solids. Even so, the levels of suspended solids exceeded existing recommendations and could cause clogging of irrigation systems. Most of the elements crossing the membranes were in soluble form and increased the electrical conductivity (EC) of the solution as a result. The solutes consisted mainly of C (57%), K (22%), N (14%, largely as ammonium ion, which accounted for 93% of all N) and Na (6%). Previous experiments with microfiltered digestates and slurries provided results similar to ours [21, 51]. The high

Table 3 Properties of the digestate liquid fraction (DLF) and maximum values recommended by international bodies or regulated by national authorities for reclaimed irrigation water

Property	Mean	SD	Max	Min	WHO	USEPA	Spain	Portugal VMR	Italy
Turbidity (NTU)	99	88	207	0.67			10 [1]		
TS (mg L^{-1})	6424	667	7099	5386					
SS (mg L^{-1})	199	166	411	31	100	30	20 [1]/35 [2]	60	10
pH	7.56	0.16	7.86	7.41	6.5–8.0	6.5–9.0		6.5–8.4	6.0–9.5
EC (dS m^{-1})	12.5	1.2	13.8	10.6	3		3 [1.2]	1	3
Total C (mg L^{-1})	4168	143	4372	3943					
Total N (mg L^{-1})	1027	98.4	1120	859	30				15
N–NO$_3^-$ (mg L^{-1})	104.2	39.4	174.8	61.0				50	
N–NH$_4^+$ (mg L^{-1})	957.7	251.0	1260.8	644.4					2.58
P (mg L^{-1})	6.63	1.18	7.75	4.34					2
K (mg L^{-1})	1596.0	192.9	1747.9	1232.2					
Ca (mg L^{-1})	48.7	10.8	66.9	37.6					
Mg (mg L^{-1})	0.117	0.01	0.132	0.101					
Na (mg L^{-1})	442.5	39.6	495.7	380.6					
SAR [(meq L^{-1})$^{1/2}$]	17.7	2.8	20.3	13.7	f(EC)		6 [1.2]	-	
B (µg L^{-1})	988	60	1089	938		750	500 [1.2]	300	1000
As (µg L^{-1})	57	6	63	46	100	100	100 [1.2]	100	20
Cd (µg L^{-1})	0	0	1	0	10	10	10 [1.2]	10	5
Co (µg L^{-1})	30	9	37	13	50	50	50 [1.2]	50	50
Cr (µg L^{-1})	17	3	19	11	100	100	100 [1.2]	100	100
Cu (µg L^{-1})	16	13	39	7	200	200	200 [1.2]	200	1000
Mn (µg L^{-1})	6	3	13	4	200	200	200 [1.2]	200	200
Mo (µg L^{-1})	12	13	40	6	10	10	10 [1.2]	5	
Ni (µg L^{-1})	110	11	123	92	200	200	200 [1.2]	500	200
Se (µg L^{-1})	17	6	24	10	20	20	20 [1.2]	20	10
V (µg·L^{-1})	10	1	11	7	100	100	100 [1.2]	100	100

TS total solids; *SS* suspended solids; *WHO* World Health Organization [78]; *USEPA* United States Environmental Protection Agency [79]; Spain, limit set by Royal Decree 1620/2007 [80]; Portugal VRM, Maximum Recommended Value (VRM) according to Republic Diary no. 176/1998 [81]; Italy, maximum limit according to Ministerial Decree no. 185/2003 [82]; f(EC), electrical conductivity function

[1] Irrigation with water in contact with fresh food for consumption

[2] Irrigation of food to be industrially processed or not intended for human consumption

Na levels, and low Ca and Mg levels, found here resulted in a very high Sodium Adsorption Ratio (SAR) that could cause structural damage in irrigated soil. In previous microfiltration experiments, the permeate swept large amounts of soluble ions such as Na$^+$, N–NH$_4^+$ and K$^+$, thereby raising EC to 19 dS m^{-1} [43] and SAR to extreme levels (86 meq L^{-1})$^{1/2}$ [52].

By effect of salts being swept, DLF contained substantial amounts of nutrients such as N and K but low levels of P. These results are consistent with previous reports. The nutrients present in DLF could be exploited by using it as fertigation water. In fact, previous experiments on pasture, corn or, in diluted form, hydroponic crops, provided similar or even better results with this type of product than with mineral fertilizers [53–55]. Some crops or management techniques

require additionally using supplements of macronutrients such as P or Mg [54, 56].

Another possible use of the water and the nutrients dissolved in DLF is as water for irrigation. In order to use it, it must comply with the reclaimed water regulations for this use. Overall, DLF would fulfil the requirements as regards suspended solids, EC, N–NO$_3^-$, total N and SAR, the latter two of which are the most restrictively regulated (Table 3). Diluting DLF may be effective to comply with existing regulatory standards. Thus, 1:35 dilution would provide reclaimed water acceptable for use in most countries. Alternatively, reverse osmosis would retain more than 95% of all Na, K and N-NH$_4^+$ present [57].

As regards heavy metals and trace elements, which are more uniformly regulated among countries, DLF only exceeded the limits for B and Mo, which are in fact plant

micronutrients and whose potential hazards can be avoided simply by 1:1 dilution.

Heavy Metals

Most heavy metals present in slurries and their digestates are in solid form, bound to organic matter, present as carbonate precipitates, bound to Fe and Mn oxides or in residual fractions [58]. Possibly as a result, removing the solid phase from the slurry reduced the contents in heavy metals relative to the starting material (Table 4). Conversely, subsequently removing the liquid fraction from the digestate, DLF, and concentrating the solids in the D and DCF increased such contents relative to SLF (Table 4). Even so, heavy metal levels remained below the thresholds set in EU Regulation (EC) 2019/1009 for different factions. Irrespective of treatment, the final DCF had heavy metal contents similar to those of the starting slurry and also to the typical values for intensive pig fattening farms [35]. By exception, the Zn content was well below average, possibly as result of its being supplied in smaller amounts through the diet. Pig slurry typically contains high levels of Cu and Zn by effect of the two metals being present in feed supplements [60]. Such high levels can result in accumulation of the metals in soil continuously receiving pig slurry or digestate [61–63].

Although the concentrations of heavy metals changed throughout, the relative weight of each metal remained constant and decreased in the following sequence, consistent with previous reports for pig slurries and digestates [35, 61, 64]: Zn > Cu > Ni > Cr > Pb > Cd > Hg.

Carbon Mineralization

The contents in mineralized C exceeded those of the control soil with all treatments. This led us to subtract the amount of C mineralized in the control soil (i.e., net mineralization) from those obtained with all other treatments. The curve for net mineralized C was fitted by using a single-pool model

and a two-pool model. As can be seen from Table 5, the SLF curve was closely fitted with both types of model. In fact, the two-pool model for SLF reports two fractions of equal magnitude that mineralize at the same rate, so it is really a one-pool. This involved assuming that mineralizable C was present in labile forms and converted into mineral forms at a high rate (0.13 day^{-1}). On the other hand, the results for C in S, D and DCF were more closely fitted by a two-pool model, which assumes mineralizable organic matter to consist of two different fractions being mineralized at a different rate and a non mineralizable fraction. Thus, there was a labile fraction accounting for 68, 84 and 65% of all mineralizable C in S, D and DCF, respectively, that was mineralized at a high rate (0.838, 1.335 and 1.175 day^{-1}, respectively) and completely degraded within 48 h. The remaining mineralizable C was bound to a more resistant fraction that was degraded at a considerably lower rate (< 0.1 day^{-1}).

The total amount of C mineralized at the end of the tests (day 112) was 32, 116, 215 and 246 mg $C\text{-}CO_2 \cdot kg$ $soil^{-1}$ with SLF, D, DCF and S respectively. These results were strongly influenced by the total amount of C supplied to the soil, which differed among treatments (Table 1).

In order to better understand the net C mineralization results, they were plotted as percentages relative to the total amount of C applied (Fig. 1). Again it is observed that, C in SLF was mineralized especially rapidly, with virtually all mineralizable carbon (73% of total carbon) being converted within 30 days. Therefore, this fraction scarcely helped maintain C levels —and hence organic matter levels— in the soil. The fast mineralization of most C can be ascribed to the most recalcitrant fraction of OM being removed as large particles together with the solid fraction, the most labile portion (soluble OM and suspended fine particles) remaining in the liquid fraction SLF. However, the labile organic portion of SLF, which consisted largely of fatty acids, was heavily degraded by anaerobic digestion being reduced in the digestate to only 14% of the total carbon. Although the digestate has less mineralizable carbon, it is very labile and

Table 4 Heavy metal contents of the starting slurry and its fractions

Metal concentration	S	SLF				D			DCF				UE limit	Spanish limit
Cr (mg kg^{-1} d.m.)	7.91	3.61	±	0.95	a	6.08	±	0.45	a	12.39	±	1.46	a	–	250
Ni (mg kg^{-1} d.m.)	10.60	6.42	±	0.72	a	6.51	±	0.26	a	7.70	±	0.21	a	50	90
Cu (mg kg^{-1} d.m.)	187.85	115.68	±	8.31	a	202.66	±	8.93	b	228.50	±	28.18	b	300	300
Zn (mg kg^{-1} d.m.)	606.88	433.23	±	104.99	a	462.08	±	19.05	a	517.63	±	57.63	a	800	500
Cd (mg kg^{-1} d.m.)	0.44	0.28	±	0.11	a	0.22	±	0.01	a	0.23	±	0.04	a	1.5	2
Hg (mg kg^{-1} d.m.)	0.31	0.17	±	0.15	a	0.21	±	0.11	a	0.22	±	0.13	a	1	1.5
Pb (mg kg^{-1} d.m.)	5.41	1.28	±	1.01	a	1.96	±	0.09	a	2.44	±	0.26	a	120	150

UE limit, highest value allowed by Regulation (EU) 2019/1009. S, slurry; *SLF* slurry liquid fraction; *D* digestate; *DCF* digestate concentrated fraction; Spanish limit, highest value allowed by Royal Decree 506/2013 for class B fertilizers [83]; d.m., dry matter. Different letters in the same row denote significant differences at $p < 0.05$

Table 5 Typical parameter values for the modelled equations for one or two carbon and nitrogen fractions

$Ci_t = C_0 \times [1 - e^{(-k \times t)}]$

	C_0	k	R
S	231.84	0.376	0.978
SLF	30.56	0.130	0.989
D	111.80	0.845	0.994
DCF	202.83	0.450	0.976

$Ci_t = C_1 \times [1 - e^{(-k_1 \times t)}] + C_2 \times [1 - e^{(-k_2 \times t)}]$

	C_1	k_1	C_2	k_2	R	
S	165.67	0.838	79.062	0.062	0.999	d
SLF	15.26	0.130	15.263	0.130	0.989	a
D	95.69	1.335	18.745	0.088	1.000	b
DCF	138.64	1.175	74.135	0.078	0.999	c

$Ni_t = Ni_0 + N_0 \times [1 - e^{(-k \times t)}]$

	Ni_0	N_0	k	R	
S	80.35	90.04	0.015	0.822	b
SLF	4.91	7.02	0.175	0.707	a
D	129.37	73.99	0.019	0.754	b
DCF	131.62	104.23	0.020	0.892	b

$Ni_t = Ni_0 + N_1 \times [1 - e^{(-k_1 \times t)}] + N_2 \times [1 - e^{(-k_2 \times t)}]$

	Ni_0	N_1	k_1	N_2	k_2	R
S	80.35	41.26	0.015	48.78	0.015	0.822
SLF	4.91	3.51	0.175	3.51	0.175	0.707
D	129.37	20.69	0.019	53.29	0.019	0.754
DCF	131.62	0	2.314	104.23	0.020	0.892

S slurry; *SLF* slurry liquid fraction; *D* digestate; *DCF* digestate concentrated fraction. Different letters in each column for a model denote significant differences at $p \leq 0.001$

its contains high levels of mineral N being readily absorbed and favouring microbial growth [65, 66]. In fact, D resulted in the greatest initial growth by effect of its containing the highest levels of labile mineralizable C. In addition, its concentrated fraction DCF and the slurry (S) gave similar curves which, however, differed in their asymptotes (i.e.,

Fig. 1 Time course of C mineralization (cumulative C-CO$_2$/total C applied) in S (squares), slurry; SLF (diamonds) slurry liquid fraction; D (triangles), digestate and DCF (crosses) digestate concentrated fraction. Experimental points and modelled curves. All modelled curves were statistically different at $p < 0.001$
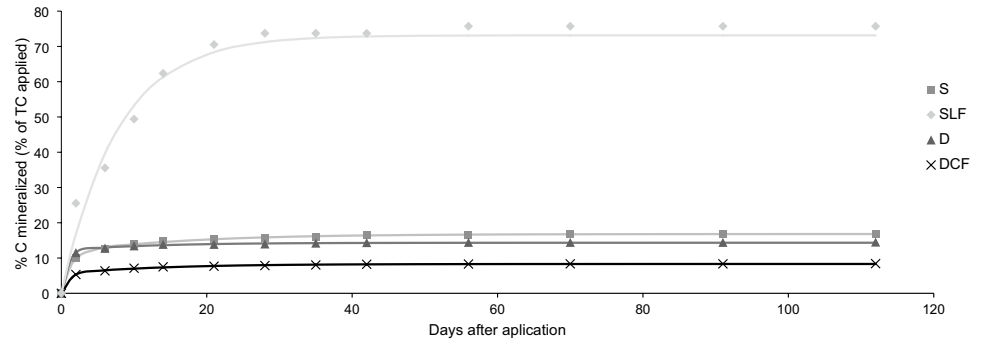
Fig. 2 Net mineralization of N (N–NO$_3^-$ + N–NH$_4^+$) in S (squares), slurry; SLF (diamonds) slurry liquid fraction; D (triangles), digestate and DCF (crosses) digestate. Experimental points and modelled curves
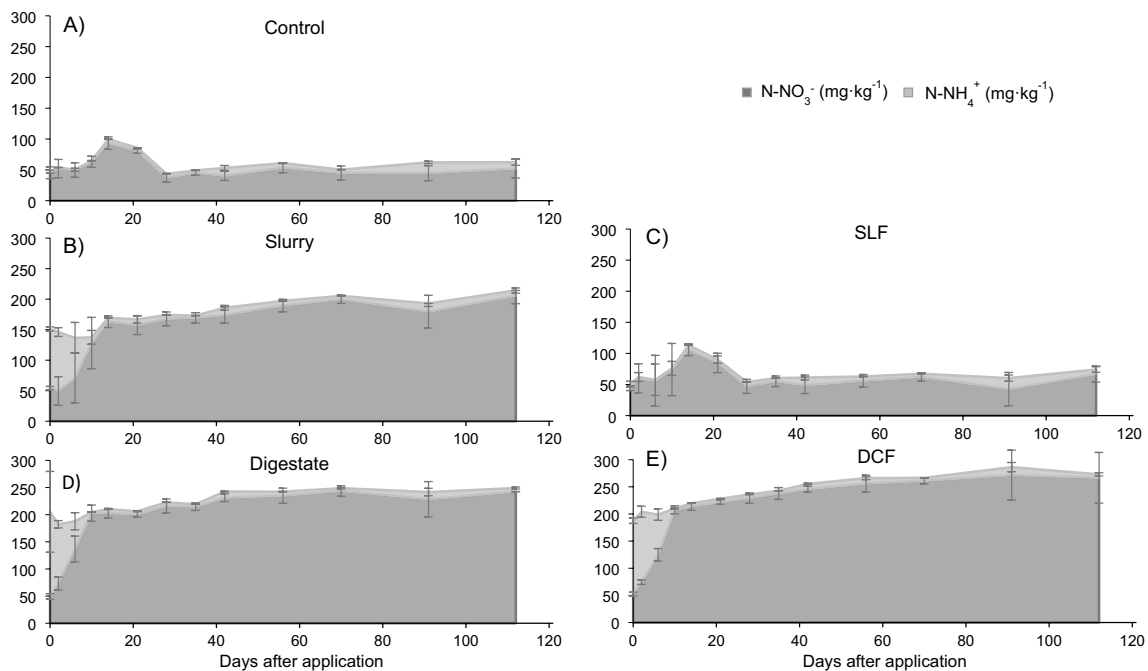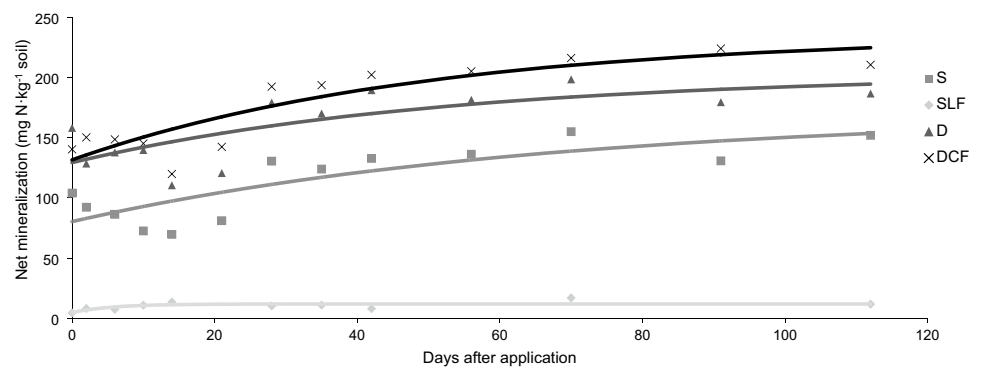
Fig. 3 Variation of the contents in nitrate (mg N–NO$_3^-$ kg^{-1}, dark grey) and ammonium (mg N–NH$_4^+$ kg^{-1}, light grey) of the soil during incubation. Bars correspond to the standard deviations of 4 replicates each. **A** Control; **B** Slurry; **C** Slurry liquid fraction; **D** Digestate. **E** Digestate concentrated fraction

in the proportions of mineralizable C). The proportion of mineralizable C in those fractions undergoing no anaerobic digestion was higher than those in the digested fractions (16.8% in S versus 73.1% in SLF). This was a result of part of mineralizable C being used for bacterial growth and biogas production during anaerobic digestion [67]. Thus, the digestate exhibited lower (14.3%) and the DCF even lower mineral C contents (8.3%) by effect of microfiltration transferring the more labile C forms to the liquid fraction.

In previous studies on slurries and their digestates, mineralizable C levels ranged widely (from 11 to 75% of total C); however, the slurries invariably had higher C contents than their digestates [65, 67, 68].

Anaerobic digestion is known to increase the relative concentration of recalcitrant substances such as lignin (especially in the digestate solid fractions), thereby increasing the proportion of non-mineralizable C in the short to medium term [68–70]. Because non-mineralizable C remains in soil over longer periods, it helps maintain OM levels. The proportion of recalcitrant C decreased in the following treatment sequence: DCF > D > S > > SLF. Consequently, anaerobic digestion of slurry does not reduce the supply of recalcitrant C to soil or diminish its C sequestration capacity. This is a consequence of most C used for anaerobic digestion would be equally mineralized and released into the atmosphere after application of the slurry [71].

Nitrogen Mineralization

Both the S, D and DCF increased the soil mineral N, to a similar extent, immediately upon application to the soil (Fig. 2). As expected, this was not the case with SLF, which contained a high proportion of moisture and hence very little N. S and D caused a slight decrease in $N-NH_4^+$ (Figs. 3B and D) and mineral N content (Fig. 2) within the first few hours after application to the soil. This is suggestive of N immobilization by effect of microbial growth as it occurred simultaneously with an increased release of $C-CO_2$ (Fig. 1)—a likely result of the increased proportions of labile C in the slurry (11% of all C) and digestate (12%) relative to DCF (5% only). Nitrogen immobilization in the presence of readily degradable C is known to result from its use by oxidative microbes [72–74]. Based on the C/N ratio of these materials immobilization was not expected; however, as some fractions of the added materials decompose faster than others, the C/N ratio of the fraction being mineralized at a certain point in time may be different from the total one [75]. De la Fuente [68] observed a similar behaviour and found net immobilization of N within one week after application of a slurry and its digestate.

As tests progressed, S, D and DCF released mineral N constantly. At the end of the trial, the mineral N content was higher in soils fertilized with DCF and D (273 and 249 mg N kg soil^{-1}, respectively) than with S (214 mg N kg soil^{-1}). In turn, these were much higher than SLF fertilized soils and the control samples (74 and 62 mg N kg soil^{-1}, respectively).

Supplying the soil with SLF, initially, slightly increased the levels of $N-NH_4^+$ (plus 6.45 mg $N-NH_4^+$), which was completely converted into $N-NO_3^-$ within 48 h, nitrate being the major ion throughout the remainder of the incubation period (Fig. 3C). In any case, SLF supplied little N to the soil, the amount of mineral N measured belonging largely to the soil itself. Also, more than 95% of all N supplied by SLF had been mineralized by day 30. All other fractions were treatment-independent. In all three, NH_4^+ was the main ion at the beginning of the tests. However, ammonium from de SLF was almost completely converted to nitrate ion within 10 days, after which the former was present at virtually residual concentrations (Fig. 3B; 3D; 3E). These results are consistent with those of Alburquerque et al. (2012) who found nearly complete nitrification of $N-NH_4^+$ from slurry digestates within the 14 first days of incubation.

Fitting the net mineral N curves revealed high correlations ($0.7 < R < 0.9$). All curves were fitted to a single-pool model because the two-pool model provided no additional advantage (Table 5). The curve for SLF departed from the others owing to the increased rate of mineralization of this fraction, all mineralizable N in which was depleted within 1 month (Fig. 2). The other fractions gave similar curves with lower rates as a result of N being mineralized more slowly —and hence over a longer period. Based on the model, the mean lifetime of mineralizable N in SLF was only 4 days, whereas those in D and DCF was 36 and 35 days, respectively, and that in S 46 days.

Based on the foregoing, S, D and DCF possess a similar potential as long-term nitrogen fertilizers since N mineralization occurs at a similar rate and from a single fraction. The most salient advantages of these N fractions (mineralizable N) is their ability to supply crops with nitrogen over long periods as a result of N mineralization and uptake by crops occurring synergistically [77].

Conclusions

Solid–liquid separation removes much of the DM, N and P from the slurry. After microfiltration, part of the concentrated digestate was recycled to the digester. This allowed to accumulate more C in it, optimizing anaerobic digestion. However, unwanted wash off of $N-NH_4^+$, K and Na into the liquid digestate fraction also occurred.

The amount of suspended solids and dissolved elements present in the liquid fraction of the digestate limited its use as reclaimed water for irrigation without prior dilution. Other post-treatments could be useful to comply with national regulations or recommendations of international organizations. Tests must be carried out to verify an alternative use as a fertigation solution.

The dynamics of the organic matter of each fraction was modified due to the process. Although in the liquid fraction of the slurry most of the C was labile (73% of the total C), after the digestion-microfiltration process it was consumed and the recalcitrant C was concentrated (91.3% of the total C).

Thanks to this, the digestate concentrated fraction could provide stable organic matter to the soil in an amount even higher than the slurry where the recalcitrant C accounted for 82.3% of the total C. Also, anaerobic digestion of the liquid fraction did not impair the C sequestration capacity of the soils.

The N dynamics of digestate concentrated fraction was similar to that of slurry, so it could continue to be used as a basal fertilizer, providing a substantial amount of immediately available N and a complementary amount of slower mineralization.

During digestion-microfiltration, the amounts of heavy metals remained similar, with the exception of Cu, which increased slightly but did not exceed the legal limits in any case.

Thus, the process, in addition to generating a widely studied solid fertilizer (solid fraction of slurry) and obtaining biogas efficiently, provided a concentrated digestate with a high fertilizer value, and regenerated water for use in dilution or as a fertigation solution.

Declarations

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Authors and Affiliations

Miguel Fernández-Labrada[1] · **María Elvira López-Mosquera[2]** · **Adolfo López-Fabal[1]**

Miguel Fernández-Labrada
miguelfernandez.labrada@usc.es

María Elvira López-Mosquera
melvira.lopez@usc.es

[1] Departamento de Producción Vegetal y Proyectos de Ingeniería, Universidade de Santiago de Compostela. Escuela Politécnica Superior de Ingeniería, Campus Universitario. 27002, Lugo, Spain

[2] Instituto de Biodiversidad Agraria y Desarrollo Rural (IBADER), Universidade de Santiago de Compostela. Escuela Politécnica Superior de Ingeniería, Campus Universitario. 27002, Lugo, Spain