Solid fraction of separated digestate as soil improver – implications for soil fertility and carbon sequestration

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ABSTRACT

Purpose
This study investigated the C and N mineralisation potential of solid fractions (SFs) from co-digested pig manure after P-stripping (P-POOR SF) in comparison with P-rich SFs, as a means to estimate their organic matter stability in soil. Compost (COMP) and biochar (BCHR) (made from P-POOR SF) were also included in the study as reference biosolids.

Methods
The SFs were incubated in a sandy-loam soil under moist conditions to determine production of CO$_2$ and mineral N. At specified intervals, CO$_2$ evolution in the mixtures was measured via the alkali trap method and titration over a period of 81 days while mineral N was measured using a flow analyser after KCl extraction over a period of 112 days.

Results
The various SFs showed similar patterns of C mineralisation (15%-26% of added total C in 81 days) that were clearly higher than for COMP and BCHR (6% and 7%, respectively). Temporary N immobilisation was observed in biosolids with a high C/N ratio. The effective organic matter (EOM) of the SFs was calculated based on the C mineralisation data and varied between 130 and 369 kg tonne$^{-1}$.

Conclusions
The SF with a reduced P content had a high EOM/P ratio which is beneficial in areas where P status of the soil is already high. Moreover, the N mineralisation patterns confirm that a high C/N ratio may also reduce risks for N leaching due to temporary N immobilisation.

Keywords: soil organic matter; solid fraction; digestate; C/N ratio; mineralization
1. Introduction

Over the last decade, European Union (EU) policy oriented towards developing a circular and resource-efficient economy has encouraged a rapid increase of anaerobic digestion (AD) installations. The number of AD installations in the EU rose from 6,227 to 17,662 between 2009 and 2016 (EBA, 2018), with a main focus on AD of residues from food and feed industries, sewage sludge, animal manure and landfill waste. While biogas is the primary product from AD, millions of tonnes of digestate are generated annually as a by-product. Digestate requires appropriate management or disposal to prevent over-fertilisation in regions with an excess of manure and digestate as well as storage problems. Digestate is typically separated mechanically into liquid and solid fractions as a simple procedure to overcome transport constraints and facilitate application to soil (Holm-Nielsen et al. 2009). The liquid fraction (LF) typically contains significant amounts of nitrogen (N), mainly in the form of plant available ammonium-N, and can be used as a N-fertiliser (Sigurnjak et al. 2017). The solid fraction (SF), on the other hand, has a high dry matter content, is rich in phosphorus (P) and organic carbon (C), and has the potential to be used as a P fertiliser and an organic soil improver.

The recently approved Fertiliser Regulation (European Commission, 2019) makes a distinction between “organic soil improvers” and “solid organic fertilisers” primarily based on the nutrient content and the intended use of the biosolid. While solid organic fertilisers are added as sources of readily available plant nutrients, organic soil improvers are added primarily as sources of organic matter (OM) in order to ameliorate soil physicochemical and biological properties. When OM is added to soil, microorganisms use it as a source of energy thereby emitting CO₂ via respiration (Juma, 1999). Hence, only the fraction of OM that is less
degradable remains and eventually contributes to soil organic matter. This stable fraction of the original OM that remains after 1 year is often referred to as the “effective organic matter” (EOM) (Veeken et al. 2017). In order to be classified as an organic soil improver, Veeken et al. (2017) proposed that the EOM to mineral-N ratio must be higher than 150 and the EOM to phosphate (P$_2$O$_5$) ratio must be higher than 35.

SFs from AD processing have a high C and P content, and so the C/P ratio may be problematic for their application as soil improvers in regions with restrictions on P input (e.g. Belgium and the Netherlands). However, these products are marketed in other countries as P-fertilisers, thus, representing a loss of organic C from such regions. A recent study has shown that up to 80% P can be recovered from SFs of digestate at reasonable costs using a P-stripper system known as Re-P-eat (Schoumans et al. 2017). This process involves addition of sulphuric acid to release the phosphate from the SF followed by adding a base to precipitate the solubilised P. Two products are then obtained – a mainly crystalline struvite (NH$_4$MgPO$_4$·6H$_2$O) and a P-POOR SF (Regelink et al. 2019; Schoumans et al. 2017). While the recovered struvite can be sold as a P fertiliser, the P-POOR SF may open opportunities for keeping C in regions with P-rich soils.

Sustaining an adequate soil organic matter content plays an important role in maintaining and improving soil microbial health and soil structure, which is crucial for attaining sustainable crop production (Chatterjee et al. 2017). Moreover, European policy has recognized soil organic carbon (SOC) as an instrument to reduce CO$_2$ emission through soil C sequestration (Lugato et al. 2014). Finally, organic soil improvers have also proven their potential utilization to reduce metal mobility in soils exhibiting elevated concentrations, and
by doing so alleviate associated environmental risks (Van Poucke et al. 2019; Egene et al. 2018). Therefore, there is a need for increased utilization of C-rich biosolids in agriculture to improve soil organic matter and increase soil C reserves.

There are a few studies that have looked at the C and N mineralisation potential of digestates in soils (Peters & Jensen, 2011; de la Fuente et al. 2013; Möller, 2015). These studies highlighted the relationships between digestate composition and C and N mineralisation dynamics. However, the effect of P-stripping of SFs of digestates on its OM decomposition and consequent C and N mineralisation has not been investigated. This makes it necessary to assess the dynamics of active C and N pools in the SFs of digestate to determine how much organic C and N will mineralise in current and future growing seasons.

The aim of this study was to evaluate the dynamics of C- and N-mineralisation in soil amended with solid digestate following different post-digestion treatments, compared to biochar and compost (as reference biosolids), in order to quantify the mineralisable C and N. To achieve this, the C and N decomposition kinetics of the different biosolids were studied through incubation experiments and were related to some of their inherent agrochemical characteristics (e.g. C/N ratio, mineral N, pH). Any such relationships should provide useful insight into the soil improving properties of SF of digestate and may be useful in estimating appropriate application rates. Furthermore, the potential of long-term application of the SFs as sources of soil organic matter in P-rich soils was evaluated.
2. Materials and methods

2.1. Biosolid collection and characterization

Six biosolids (Table 1) were used: four SFs that went through different post-digestion treatment steps, biochar and compost. All SFs were collected in 2018 from full scale AD installations of either Groot Zevent Vergisting (GZV) (Beltrum, Netherlands) or AM Power (Pittem, Belgium). Briefly, the AD plant of AM Power has a total utilisable volume of 20,000 m³ producing 7,360 and 7,435 kWh day⁻¹ of thermal and electrical energy, respectively, by the anaerobic digestion of organic biological waste. GZV is an AD plant with a volumetric capacity of 15,000 m³ producing 10 Mm³ of biogas by mesophilic digestion of animal manure (74%) and various organic waste products from the agro-food industry plus glycerin (26%) as co-substrates.

In this study, the SFs are differentiated primarily based on the type of post-digestion treatment(s) of the digestates. The digestate from AM Power underwent solid-liquid separation with a decanter centrifuge after which the SF was passed through a fluidized bed dryer at 60 °C to obtain a dried SF denoted as DRY-SF. Three SFs were obtained from the digestate of GZV: (i) SF separated by a decanter centrifuge (DEC-SF) (ii) SF separated by a screw press (SCP-SF) and (iii) SF separated by a decanter centrifuge followed by P-stripping (P-POOR SF). The P-stripping process, called Re-P-eat, was developed by Wageningen UR and has been demonstrated at pilot scale (Schoumans et al. 2017). The process involves acidifying (H₂SO₄) the SF of digestate to pH 5 to solubilize mineral P followed by the solid-liquid separation of the acid slurry to obtain a P-rich acid solution and a P-poor SF. Ca(OH)₂ or Mg(OH)₂ is then added to the acid solution to precipitate the P as calcium phosphate or struvite (Schoumans et al. 2017).
Biochar (BCHR), produced via slow pyrolysis of the P-POOR SF at 400 °C, and was included in the study. In addition, commercial compost (COMP) produced by Attero (Venlo, The Netherlands) from source-separated household and garden waste was used as reference material.

All biosolids were collected in polyethylene sampling bottles and stored at 4 °C. The dry matter (DM) content was determined by drying to constant weight (48 h) at 80 °C and was calculated as percentage of wet weight. OM was measured based on loss-on-ignition (LOI) of dried solids by incineration at 550 °C in a muffle furnace for 4 h. The pH and electrical conductivity (EC) were measured potentiometrically using an Orion-S20A pH-meter and a WTW-LF537 (GE) conductivity electrode, respectively, in a 1:5 (w/w) wet solids to deionised water ratio. Total C, N and Sulphur (S) were determined via Elemental analysis (Variomax CNS analyzer, Elementar, Germany). Ammonium (NH₄⁺-N) and nitrate (NO₃⁻-N) were analysed in a 1:10 (w/w) suspension of wet solid and 1 M KCl shaken end-over-end for 30 min. The extracts were filtered (Whatman No. 45) and analysed for their NH₄⁺-N and NO₃⁻-N contents with a continuous flow auto-analyser (Chemlab System 4, Skalar, the Netherlands).

Total P, potassium (K), calcium (Ca), magnesium (Mg) and sodium (Na) were determined using an inductively coupled plasma emission spectrometry (ICP-OES) (Varian Vista MPX, USA) after wet digestion of the incinerated solids with 5 mL of 6M HNO₃ and 5 ml of 3M HNO₃ at 150 °C (Van Ranst et al. 1999). Water soluble carbon (WSC) and hot water extractable carbon (HWEC) were determined on fresh samples according to a modified method of Ghani et al. (2003). For WSC, 3 g of each biosolid and 30 ml of distilled water was weighed into 50 ml polypropylene centrifuge tubes, which were shaken end-over-end for 30
minutes. The tubes were centrifuged for 20 min at 3500 rpm and the supernatants were filtered (Whatman No. 45), and transferred into vials for C analysis. To determine the HWEC, 30 ml of distilled water was added to the residues from the previous step, shaken briefly to re-suspend the solids, capped well and placed in a hot water bath at 80 °C for 16 h. The extracts were centrifuged for 20 min at 3500 rpm, filtered (Whatman No. 45), and transferred into vials for C analysis. Total C (inorganic and organic C), in both the first and second extracts, was determined using a TOC-analyser (TOC-5000, Shimadzu, Tokyo Japan).

2.2 Soil characteristics

Soil for the incubation experiments was taken from the topsoil layer (0-30 cm) of an unfertilised agricultural field in Evergem (Belgium). The soil texture was sandy-loam (87% sand, 8.5% silt and 4.5% clay), with a pH-H₂O of 7.1, initial C content of 118 mg kg⁻¹, and an initial exchangeable N content of 1.7 mg NH₄⁺-N kg⁻¹ and 21 mg NO₃⁻-N kg⁻¹. Prior to incubations, the soil was air-dried and sieved through a 2-mm screen.

2.3 C incubation experiment

Organic C mineralisation was evaluated by measurement of soil CO₂ respiration (De Neve & Hofman, 2000). First, the soil was wetted with distilled water to attain a moisture content, corresponding to a 50% water filled pore space (WFPS), taking into account the moisture content of the air-dried soil and tested organic materials. The tested organic materials were mixed with 117 g of soil at an application rate of 9000 kg OC ha⁻¹ (or 28 g C kg soil⁻¹). These mixtures were filled into PVC tubes (7.2 cm height, 6.8 cm diameter) and gradually compacted during filling to attain an apparent bulk density of 1.4 g cm⁻³. The conversion
The percentage of WFPS in the soil was calculated from the equation (Linn and Doran 1984):

\[
WFPS\% = \left(\frac{GWC \times BD}{1 - \frac{BD}{PD}}\right) \times 100
\]

Equation (1)

where GWC is the gravimetric water content (g water g soil\(^{-1}\)), BD is the dry bulk density (Mg m\(^{-3}\)), and PD is the particle density (Mg m\(^{-3}\)). The PVC tubes containing the soil-biosolid mixtures were placed in 1 L Mason jars and just before closing them airtight, glass vials containing 15 ml 1 M NaOH were placed to capture CO\(_2\) evolved. Controls were performed without soil samples and with unamended soil. The incubation was done under controlled conditions at 10 °C for 81 days. At predetermined sampling times (1, 2, 3, 5, 9, 14, 21, 28, 36, 45, 59 and 81 days), the glass vials were removed for determination of amounts of evolved CO\(_2\) by titration of the NaOH with 1 M HCl after precipitation of carbonates with excess BaCl\(_2\) (Anderson, 1982). During each sampling time, moisture was adjusted to 50% WFPS and the jars were left opened for at least 2 hours to allow replenishment of oxygen.

### 2.4 N incubation experiment

The evolution of N mineralisation was determined by measuring temporal changes in the mineral N (NH\(_4\)\(^{+}\)-N and NO\(_3\)\(^{-}\)-N). The air-dried and sieved soil was brought to 35% of WFPS and was pre-incubated for 1 week at 21.5 °C. This step was necessary to stabilize the microbial community in the soil. After pre-incubation, each of the biosolids was applied at a rate of 9000 kg OC ha\(^{-1}\) (as in the C incubation) and thoroughly mixed with approximately 272 g of pre-incubated soil. Table 2 shows the corresponding total N, organic N and mineral N dosages. The soil-biosolid mixtures were placed in PVC tubes (18.2 cm height, 4.61 cm diameter), again, at a bulk density of 1.4 g cm\(^{-3}\) and the WFPS in the tubes was brought up to 50% by adding distilled water. In total, 256 PVC tubes (8 treatments x 4 replicates x 8
sampling times) were covered with pin-holed gas permeable parafilm, placed on trays in a completely randomized design (CRD) and incubated for 112 days at 21.5 °C. Destructive sampling was done bi-weekly in which 32 intact tubes (4 treatments x 4 replicates) were removed. After mixing well, 10g of soil per tube was analyzed for its mineral N contents as described in 2.1. The sum of soil NH$_4$-N and NO$_3$-N build-up in excess to the unamended soil was considered as the amount of N mineralised per sampling time.

2.5 Data and statistical analysis

The cumulative amount of mineralised C, evolved as CO$_2$ (cumulative % C mineralised) from each of the tested biosolids was calculated as the difference between the cumulative CO$_2$-C evolved from the unamended (control) soil and the biosolid-treated soil. Cumulative amounts of mineralised C were expressed as a percentage of the amount of TOC applied (Equation 2) and is hereafter referred to as %C mineralised (%C$_{\text{min}}$).

$$\%C_{\text{min}} = \frac{(C_{\text{min, treatment}} - C_{\text{min, control}})}{\text{TOC applied}} \times 100$$  \hspace{1cm} \text{Equation (2)}$$

A second order kinetic model was fitted to the %C$_{\text{min}}$ data for each of the tested biosolid using the Grapher Statistical software (Version 10). The second order kinetic model (Equation 3) suggests that the mineralisation or decomposition is proportional to the product of the substrate concentrations and of the microorganisms derived from the substrate (Sleutel et al. 2005) and is expressed by:

$$C(t) = C_A - \frac{C_A}{1 + K_a (1-a) C_A t}$$  \hspace{1cm} \text{Equation (3)}$$
Where \( C(t) \) is the cumulative amount of C mineralised at time \( t \), \( C_A \) is the amount of mineralisable C, \( \alpha \) is the fraction of decomposed substrate that becomes part of microbial biomass, and \( k_2 \) is the second order C-mineralisation rate constant. As a simplification, \( k_2 \) and \( \alpha \) were determined together as a single variable \( k_2\alpha(1-\alpha) \). The model parameters were then used to extrapolate the stable organic fraction left in the soil after a period of 365 days (i.e. the humification coefficient).

Net N mineralisation (Equation 4) in the biosolid-amended treatments and unamended control was calculated by subtracting the mineral N (\( N_{\text{min}} \)) content of the treatment at day 0 from the \( N_{\text{min}} \) in the sample at all subsequent measurements and expressed as percentage of organic N (\( N_{\text{org}} \)) and total N (\( N_{\text{tot}} \), as follows:

\[
\begin{align*}
\text{Net mineralisation: (t; % organic N)} & = \frac{N_{\text{min}}(t=x) - N_{\text{min}}(t=0)}{N_{\text{org}}} \\
\text{(t; % total N)} & = \frac{N_{\text{min}}(t=x) - N_{\text{min}}(t=0)}{N_{\text{tot}}}
\end{align*}
\]

Equation 4

Treatment effects were compared using one-way ANOVA and the Tukey’s honestly significant difference (HSD) test. Pearson’s correlation analysis was performed to test relationships between variables. All tests were carried out at a probability (\( P \)) level of 0.05 using SPSS 22.0 software for Windows.
3. Results

3.1. Biosolid characteristics

The agrochemical characteristics of the tested biosolids are summarized in Table 3. With the exception of P-POOR SF, the pH of the tested biosolids were alkaline (7.3-8.5) which is consistent with previously reported pH values of digestate and digestate products (Alburquerque et al. 2012; Pognani et al. 2009). The acidic pH of the P-POOR SF (5.7) was attributed to the P-stripping process that involved the addition of acid (H₂SO₄). Pyrolysis of the P-POOR SF to produce BCHR greatly altered its agrochemical properties, with a noticeably, higher pH, total carbon (TC) and total nitrogen (TN) contents (Table 3).

TC, TN and inorganic N content in the solid fractions were variable. DRY-SF had the highest TC (275 g kg⁻¹) and TN (30 g kg⁻¹) while DEC-SF had the lowest TC (102 g kg⁻¹) and SCP-SF the lowest TN (5.0 g kg⁻¹). This variability is reflected in the C/N tot ratios which ranged between 9.2 and 36 (Table 3). The tested biosolids were generally characterized by very low amounts of NO₃⁻-N. The NH₄⁺-N/TN ratio, which provides an indication of the amount of readily plant-available N, was low in all the tested biosolids. It was lowest in DRY-SF and BCHR due to drying and pyrolysis, respectively, which are processes that result in a significant loss of NH₄.

Moderate amounts of TN was present in the SFs, mostly in the form of organic N, as could be expected since the solid-liquid separation of digestate concentrates the major part of the mineral N in the LF. In general, their OM and N contents did not show much variability (74–89% OM, 5.0–5.7 g kg⁻¹ TN). However, high variability was observed in the total P content with DEC-SF having 1.7 and 5 times more P than SCP-SF and P-POOR SF, respectively. DRY-SF
was characterized by a relatively low OM content (59%) and higher concentration of nutrients, including TN (30 g kg\(^{-1}\)) and TP (14 g kg\(^{-1}\)), compared to the other SFs.

The WSC and HWEC, which have been reported to constitute the highly labile pool of C in organic materials (Ghani et al. 2003; Weigel et al. 2011), represented < 2% and ≤ 3%, respectively, of the total C in all the tested biosolids. This suggests that majority of the C in were present as the stable carbon fraction.
3.2. C mineralisation

The C_{min} of the tested biosolids was calculated according to Equation 2. The results were expressed in terms of the percentage of C added and mineralisation curves were plotted (Fig. 1). At the end of the incubation period, the highest C_{min} was observed in SCP-SF (23%) at 5% significance level. The C_{min} of DEC-SF, DRY-SF and P-POOR SF did not significantly (P<0.05) differ from each other but were significantly higher than in COMP (6%) and biochar (4%). In general, the curves indicate that the mineralisation of the SFs followed similar patterns that clearly differed from the compost and biochar treatments.

The %C_{min} curves for the different biosolids were fitted to a second-order kinetic model (Equation 3) with high coefficients of determination (Table 4). The amount of mineralisable C (C_A) followed the same pattern of C_{min}: SCP-SF>DEC-SF>P-POOR SF>DRY-SF>BCHR>COMP.

Humification coefficients (HC), which is defined as the percentage of OC that remains after one year, was calculated for each biosolid using the second order kinetic model. Among the SFs, SCP-SF had the lowest HC (51%) which indicates that it contained the least stable forms of OC, while other SFs contained the more stable forms of OC with HCs between 70 and 75 (Table 4). The variation in HC may be explained by differences in C_A in the tested biosolid which showed a positive linear relationship between HC and C_A (r=0.99, P<0.05). The HC of compost in this study (93%) was in agreement with values (95 and 93%) reported by De Neve et al. (2003) and Postma & Ros (2016), respectively. Table 6 shows that HC was positively correlated with N_{org}/N_{tot} ratio (r=0.82, P<0.05) and negatively correlated with N_{min}/N_{tot} ratio (r=-0.82, P<0.05).
3.3. Estimating carbon retention potential

The humification coefficients (HC) can be expressed in kg tonne\(^{-1}\) of DM to derive the effective organic matter (EOM), which is defined as the OM that is left in the soil after 365 days (Veeken et al. 2017). Among the solid digestates tested, the highest EOM calculated was in DRY-SF at 369 kg tonne\(^{-1}\) (Table 5). This means that after an initial application of 1 tonne of biosolid, 369 kg of OM will remain in the soil after one year.

The OM/P ratio which gives an indication of the quantity of biosolid that can be applied within P limits was also determined (Table 5). DRY-SF had an OM/P ratio of 36 kg kg\(^{-1}\); of which 27 kg kg\(^{-1}\) was EOM. For the P-POOR SF instead, the EOM/P ratio was 204 kg kg\(^{-1}\) meaning that 7.5 times more EOM per kg of added P can be applied to soil as compared to DRY-SF.

3.4. N-mineralisation

At the start of the incubation, the mineral N in all treatments was present mainly as NH\(_4\)-N (Fig. 2a). However, by the 14\(^{th}\) day, majority of the NH\(_4\)-N was already nitrified (Fig. 2b). NO\(_3\)-N increased steadily throughout the duration of the incubation and was highest in DRY-SF and DEC-SF with 129 and 113 mg kg\(^{-1}\), respectively after day 112. The pattern of total mineral N evolution and that of NO\(_3\)-N were similar since increase in NH\(_4\)-N concentration were negligible.

N mineralisation was calculated as a percentage of the N\(_{org}\) present in the tested biosolid (Fig. 3). N immobilisation occurred in BCHR, P-POOR SF and SCP-SF while DEC-SF, DRY-SF and COMP yielded modest near-linear N mineralisation patterns during the incubation, with the
highest $N_{org}$ mineralisation of 28% (in DEC-SF). N is more likely to be net mineralised from substrates with low C/N ratio while substrates with C/N ratio > 20 tend to cause net N immobilisation in soil (Mendham et al. 2004; Muhammad et al. 2011; Nicolardot et al. 2001; Wagner & Wolf, 1999). This was reconfirmed in this study, where $%N_{org}$ mineralisation and $C/N_{tot}$ ratio were strongly negatively correlated ($r=-0.88, P<0.05$) (Table 6). The high immobilisation of N from SCP-SF was likely due to its high $C/N_{org}$ ratio (76) in combination with its high NH$_4^+$-N that was readily available for fast initial microbial immobilisation (Calderón et al. 2005). P-POOR SF incorporation also induced temporary net N immobilisation in the first 56 days of incubation, and this may also be attributed to the high $C/N_{org}$ ratio (32) of this biosolid. The mineralisation patterns of SCP-SF and P-POOR SF, however, do suggest a re-mineralisation of the immobilised N.
4. Discussion

4.1. Agrochemical characteristics of tested biosolids

The high alkalinity of the solid fractions (with the exception of P-POOR SF) may influence soil pH and nutrient bioavailability when added to soil. For example, nitrification in soil is highest at pH between 7.5–8.0 while ammonification is optimal between pH 6.0-8.0 (Wood 1988).

Although DEC-SF, SCP-SF and P-POOR SF were obtained from digestates derived from the same digested feedstock of GZV (Table 1), they showed a large variation in their total P content. This suggests that the type of post-digestion treatment (i.e. solid-liquid separation technique, P-stripping) may have a stronger influence on P composition of the SFs than on other nutrient parameters. P-POOR SF not only had the lowest P content but also the lowest K concentration. This was because leaching of P also removes K due to their similar mobility kinetics (Nishanth & Biswas, 2008).

The loss of moisture from drying resulted in increased concentration of nutrients in DRY-SF. Although the mineral N content was low (2.0 g kg⁻¹), the high organic N level may lead to a steady supply of N to crops provided there would be continuous mineralisation. These characteristics suggest that DRY-SF is more suited for use as a solid organic fertiliser rather than as organic soil improver. Furthermore, its high DM content (84%) may ease the storage and transportation of the DRY-SF.

There was no significant correlation between either WSC or HWEC and %Cmin (r=0.11 and r=0.25, respectively; P<0.05). For instance, the WSC and HWEC were highest in compost even though %Cmin in compost was very low. This may be due to the inherent heterogeneity of the feedstocks as well as differences in processing technologies applied. These results suggest that
WSC and HWEC may not be not good indicators of labile OM, at least, for the biosolids investigated in this study.

4.2. C and N mineralisation

The incorporation of the different biosolids significantly modified the C and N dynamics of the soil. Some studies (Flavel & Murphy, 2006; Wang et al. 2004) have shown that dissimilarities in decomposition rates (i.e. %Cmin) may be due to differences in the nature of organic carbon compounds present in the substrates. That is, mineralisation patterns in soils are mainly influenced by the initial composition of the organic material (Heal et al. 1997; Aerts, 1997; Teklay et al. 2007). For example, the C/N$_{tot}$ ratio may influence substrate decomposition rate such that substrates with a high C/N$_{tot}$ ratio above 25 are poorly decomposable (Nicolardot et al. 2001). In this study, however, SCP-SF had the highest C/N$_{tot}$ ratio (36) and yet showed fast degradability. This was likely caused by both higher C and N availability, which was reflected by the lower WSC/WHEC to N$_{min}$ ratio (Table 3). Such an occurrence will favour the preferential utilisation of labile biosolid-derived C by soil microorganisms and cause high C mineralisation. The corollary effect of high C mineralisation in SCP-SF treatment was the rapid N immobilisation observed in the first 28 days of the N incubation experiment (Fig. 3).

To an extent, P-POOR SF showed similar behaviour as SCP-SF but with moderate C mineralisation and less rapid N immobilisation. N mineralisation in P-POOR-SF was inhibited at the beginning but caught up to the unamended control at around Day 70, indicating N was likely immobilised by soil microorganisms and then re-released due to microbial turnover. In addition, the low pH of P-POOR-SF may have slowed down the turnover of microbial biomass and extended the microbial immobilisation as (a) low pH slows down bacterial cell division.
(Bååth et al. 1998) and (b) low pH favours the prevalence of soil fungi with longer life-spans (Gyllenberg and Eklund 1974). It is important to note that the mineralisation patterns of SCP-SF and P-POOR SF do suggest a re-mineralisation of the immobilised N. Therefore, both biosolids may not be suitable for supplying N to crops grown during the season of application but can serve as an N source in the subsequent growing season.

The amendment of DEC-SF induced similar amount of C mineralised (Fig. 1) but greater N mineralisation when compared to DRY-SF (Fig. 1 and Fig. 2c). This observation may be explained by the "microbial N mining" theory (Craine et al. 2007; Moorhead & Sinsabaugh, 2006) which postulates that N deficiency can enhance microbial decomposition of labile substrates in order to obtain N from recalcitrant OM. That is, the relatively higher C/N\textsubscript{tot} ratio in DEC-SF might have driven the soil microorganisms to decompose more N-rich OM for their N requirement when compared to DRY-SF where N was not limited. Considering their continued net mineralisation of organic N (Fig. 3), DEC-SF and DRY-SF appears to hold the highest N fertiliser value. Given the rather slow pace of N release, their intended use should probably be to fertilise the soil rather than the crop, as practiced in organic farming, in order to improve soil quality and fertility (Webb et al. 2010).

Despite its low C/N ratio, the C mineralisation of compost progressed slowly (6% after 81 days) indicating it to be more stable in soil than the digestate SFs here tested. That is because the undecomposed C in compost exists in complex forms that is inherently resistant to decomposition (Sana and Soliva, 1987; Robertson & Groffman, 2006). Similarly, the C in BCHR is recalcitrant, but it is not inert and can be slowly mineralised (Singh & Cowie, 2014; Zimmerman, 2010). BCHR had a high C/N\textsubscript{org} ratio and yet showed very little N immobilisation. This was
attributed to its high humification coefficient (91%) meaning that is no need for mineral N to sustain microbial growth.

4.3. Implications for using solid digestates as an organic soil improver

The relative proportions of TOC mineralised from the tested biosolids reflect their different biostabilities in soil. Estimates of humification coefficients (HC) and effective organic matter (EOM) are good indicators of long term availability of OM in soil after initial application of biosolids. The high EOM of DRY-SF (369 kg tonne⁻¹) represents a significant potential source of soil organic matter. However, its use as an organic soil improver may be limited because restrictions on P input in some EU regions (e.g. Belgium, the Netherlands and Denmark) constrain the application of P-rich materials (Amery & Schoumans, 2014). The P-POOR SF, with its high OM/P proves to be far more advantageous in such regions. In fact, with the exception of BCHR, P-POOR SF can be applied to soil at least 2.3 times more than any of the other biosolids tested (Table 5). Furthermore, its low N-content permits the application of large amounts without violating the European Nitrates Directive (91/676/EEG; European Commission, 1991). The slow N mineralisation of P-POOR SF also makes it ideal for use as an exclusively organic soil improver in regions with restrictions on P and N additions to soils.

P-stripping, as an additional treatment step, may thus be a promising technique for valorisation of SFs of digestate. Pyrolysis of the SFs (of P-POOR SF in this study) increased OM stability but still appears unwarranted to promote pyrolysis as a sustainable post-treatment, especially considering the high production costs involved (Campbell et al. 2018). Composting of SFs has been proposed by some studies as a viable alternative to increase its biological stability (de la Fuente et al. 2013; Torres-Climent et al. 2015). However, a study carried out by Tambone et al.
(2015) showed that composting did not remarkably increase the stability of SFs and Möller (2015) argued that digestate composting may reduce the fertiliser’s nutrient value and may cause greenhouse gas emissions. Given the already high stability of the tested SFs, except for SCP-SF, it again seems likely that composting would not further valorise the SF as a soil improver and the extra economic costs involved may be unfavourable. In any case, the here observed differences in the composition and mineralisation rates of SFs from digestate processing highlight the need for detailed analyses before using them as soil amendments.
5. Conclusions

The C and N mineralisation potential of P-POOR SF was compared with conventional SFs of digestate to study the stability of the present OM. The results showed that the humification coefficient does not correlate with the C/N ratio, HWEC or WSC. It therefore seems that the nature of the organic matter was the main factor controlling C mineralisation in the treatments. Temporary N immobilisation was observed in P-POOR SF and SF after screw press (SCP-SF) and this was attributed to their high C/N$_{org}$ ratios. In biochar (BCHR), no N immobilisation was observed despite its high C/N$_{org}$ ratio which is due to the high humification coefficient (91%) of the biosolid meaning that no need for mineral N to sustain microbial growth. The other tested biosolids, SF after decanter (DEC-SF), SF after decanter and drying (DRY-SF) and compost, showed a near linear positive N mineralisation. There was no correlation between the HC and %N mineralisation. When considering the amount of effective organic matter (EOM) that can be applied to soils within the phosphate limits, i.e. the EOM/P ratio, P-POOR SF had the highest potential to be used as an organic soil improver among the SFs. As such, reducing the P concentration in SFs facilitates their application in many European soils. These results highlight the agricultural value of SFs of digestate for soil organic matter, and their potential limitations in P-rich soils.
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### Table 1. Description of the studied organic materials

| Product | Name       | Feedstock                          | Post-digestion treatment at AD plant | Solid-liquid separation | Additional treatment               |
|---------|------------|------------------------------------|--------------------------------------|-------------------------|------------------------------------|
| DEC-SF  | Solid fraction | 74% pig manure and 26% co-substrates | Decanter centrifuge                   | -                       |                                    |
| SCP-SF  | Solid fraction | 74% pig manure and 26% co-substrates | Screw press                          | -                       |                                    |
| P-POOR SF | Solid fraction | 74% pig manure and 26% co-substrates | Decanter centrifuge                   | P stripping (Re-P-eat)   |                                    |
| DRY-SF  | Solid fraction | Organic biological waste           | Decanter centrifuge                   | Drying at ~60 °C        |                                    |
| COMP    | Commercial compost | Source-separated household and garden |                                      |                         |                                    |
| BCHR    | Biochar     | P-POOR SF                          |                                      | Pyrolysis at 400 °C     |                                    |

DEC-SF: solid fraction after decanter, SCP-SF: solid fraction after screw press, P-POOR SF: solid fraction decanter and P stripping.

DRY-SF: solid fraction after decanter and drying, COMP: compost, BCHR: biochar produced from P-POOR SF.
Table 2. Dosages in kg ha\(^{-1}\) for total organic carbon (TOC), total N (N\(_{\text{tot}}\)), organic N (N\(_{\text{org}}\)) and mineral N (N\(_{\text{min}}\)) of the tested products as implied in the C incubation and N mineralisation experiment.

| Product     | TOC  | N\(_{\text{tot}}\) | N\(_{\text{org}}\) | N\(_{\text{min}}\) |
|-------------|------|--------------------|--------------------|------------------|
| DEC-SF      | 9000 | 500                | 306                | 194              |
| SCP-SF      | 9000 | 252                | 118                | 134              |
| P-POOR SF   | 9000 | 309                | 283                | 26               |
| DRY-SF      | 9000 | 983                | 763                | 220              |
| COMP        | 9000 | 788                | 776                | 12               |
| BCHR        | 9000 | 316                | 316                | 0.42             |

DEC-SF: solid fraction after decanter, SCP-SF: solid fraction after screw press, P-POOR SF: solid fraction decanter and P stripping, DRY-SF: solid fraction after decanter and drying, COMP: compost, BCHR: biochar produced from P-POOR SF.
### Table 3. Agrochemical characteristics of the tested organic materials (n = 3 ± standard deviations)

| Parameter                      | DEC-SF       | SCP-SF       | P-POOR SF    | DRY-SF       | COMP         | BCHR         |
|--------------------------------|--------------|--------------|--------------|--------------|--------------|--------------|
| **pH**                         | 8.4 ± 0.1    | 8.5 ± 0.0    | 5.7 ± 0.0    | 7.8 ± 0.1    | 8.5 ± 0.0    | 7.3 ± 0.0    |
| **EC (mS cm⁻¹)**               | 2.4 ± 0.2    | 1.1 ± 0.0    | 1.4 ± 0.0    | 6.1 ± 0.0    | 0.7 ± 0.0    | 2.8 ± 0.0    |
| **DM (%)**                     | 25 ± 0.6     | 37 ± 0.4     | 34 ± 0.1     | 84 ± 0.7     | 54 ± 0.4     | 98 ± 0.2     |
| **OM (%) of DM**               | 74 ± 2.9     | 89 ± 0.2     | 89 ± 0.1     | 59 ± 1.5     | 30 ± 0.8     | 75 ± 0.0     |
| **TOC (g kg⁻¹)**               | 102 ± 4.0    | 177 ± 2.2    | 161 ± 0.9    | 275 ± 4.3    | 70 ± 2.5     | 643 ± 0.2    |
| **WSC (% of TOC)**             | 1.7 ± 0.0    | 0.9 ± 0.1    | 0.3 ± 0.0    | 0.8 ± 0.1    | 1.7 ± 0.1    | 0.04 ± 0.0   |
| **HWEC (% of TOC)**            | 3.0 ± 0.3    | 1.0 ± 0.1    | 0.6 ± 0.0    | 1.2 ± 0.0    | 4.3 ± 0.2    | 0.05 ± 0.0   |
| **Nₜₜ (g kg⁻¹)**               | 5.7 ± 0.1    | 5.0 ± 0.1    | 5.5 ± 0.2    | 30 ± 0.1     | 6.1 ± 0.2    | 23 ± 0.1     |
| **Nₘₘ (g kg⁻¹)**               |              |              |              |              |              |              |
| NH₄⁺-N                         | 2.2 ± 0.1    | 2.7 ± 0.0    | 0.46 ± 0.0   | 2.0 ± 0.0    | 0.22 ± 0.0   | 0.03 ± 0.0   |
| NO₃⁻-N                         | 0.002 ± 0.0  | 0.003 ± 0.0  | 0.001 ± 0.0  | 0.1 ± 0.0    | 0.01 ± 0.0   | 0.001 ± 0.0  |
| C/Nₜₜ                          | 18           | 36           | 29           | 9.2          | 11           | 28           |
| C/Nₘₘ                          | 29           | 76           | 32           | 9.8          | 12           | 28           |
| Nₘₘ/Nₜₜ (%)                    | 39           | 54           | 8.0          | 7.0          | 2.0          | 0.0          |
| **Elements (g kg⁻¹)**           |              |              |              |              |              |              |
| P                              | 5.6 ± 0.2    | 3.3 ± 0.1    | 1.1 ± 0.0    | 14 ± 1.1     | 1.7 ± 0.2    | 3.1 ± 0.2    |
| Ca                             | 5.8 ± 0.8    | 4.1 ± 0.4    | 3.4 ± 0.6    | 18 ± 1.1     | 12 ± 0.8     | 12 ± 1.0     |
| Mg                             | 3.5 ± 0.5    | 1.7 ± 0.3    | 0.7 ± 0.2    | 3.5 ± 0.2    | 1.7 ± 0.1    | 9.1 ± 1.2    |
| K                              | 3.2 ± 0.3    | 2.3 ± 0.2    | 0.8 ± 0.0    | 9.2 ± 0.7    | 2.4 ± 0.1    | 11 ± 0.2     |
| Al                             | 0.93 ± 0.1   | 0.34 ± 0.1   | 0.35 ± 0.0   | 2.8 ± 0.2    | 3.2 ± 0.4    | 1.6 ± 0.0    |

EC: electrical conductivity, DM: dry matter, OM: organic matter, TOC: total organic carbon, WSC: water soluble carbon, HWEC: hot water extractable carbon, Nₜₜ: total nitrogen, Nₘₘ: mineral nitrogen, Nₘₘ: organic nitrogen, DEC-SF: solid fraction after
decanter, SCP-SF: solid fraction after screw press, P-POOR SF: solid fraction decanter and P stripping, DRY-SF: solid fraction after decanter and drying, COMP: compost, BCHR: biochar produced from P-POOR SF.
Table 4. Kinetic C-mineralisation parameter values and fit, and humification coefficients (HC) for the tested products at 10 °C. Parameter data obtained with a second order kinetic model (Equation 3).

| Parameters | DEC-SF | SCP-SF | P-POOR SF | DRY-SF | COMP | BCHR |
|------------|--------|--------|-----------|--------|------|------|
| $C_a$      | 36.27  | 67.44  | 34.29     | 29.05  | 7.065| 9.793|
| $K_a(1-a)$ | 3.57x10^{-4} | 9.62x10^{-5} | 3.16x10^{-4} | 5.33x10^{-4} | 6.30x10^{-3} | 2.38x10^{-3} |
| $R^2$      | 0.997  | 0.989  | 0.999     | 0.998  | 0.955| 0.982|
| HC (%)     | 70     | 51     | 73        | 75     | 93   | 91   |

DEC-SF: solid fraction after decanter, SCP-SF: solid fraction after screw press, P-POOR SF: solid fraction decanter and P stripping.

DRY-SF: solid fraction after decanter and drying, COMP: compost, BCHR: biochar produced from P-POOR SF.
### Table 5. Composition of organic fertilising products in kg per tonne product

| Parameter                        | DEC-SF | SCP-SF | P-POOR SF | DRY-SF | COMP | BCHR |
|----------------------------------|--------|--------|-----------|--------|------|------|
| DM (kg tonne⁻¹)                  | 251    | 365    | 335       | 840    | 535  | 982  |
| OM (kg tonne⁻¹)                  | 185    | 324    | 298       | 492    | 160  | 737  |
| HC (%)                           | 70     | 51     | 73        | 75     | 93   | 91   |
| EOM (kg tonne⁻¹)                 | 130    | 165    | 217       | 369    | 148  | 671  |
| P (kg tonne⁻¹)                   | 5.6    | 3.3    | 1.1       | 13.7   | 1.7  | 3.0  |
| OM/P (kg kg⁻¹)                   | 33     | 97     | 279       | 36     | 93   | 244  |
| EOM/P (kg kg⁻¹)                  | 23     | 49     | 204       | 27     | 86   | 222  |
| N mineralisation                 |        |        |           |        |      |      |
| as % of Nₜot                      | 22     | -7     | -6        | 25     | 15   | 5    |
| as % of Nₜorg                     | 81     | -13    | -7        | 27     | 15   | 7    |

DM: dry matter, OM: organic matter, HC: humification coefficient, EOM: effective organic matter, Nₜot: total nitrogen, Nₜorg: organic nitrogen, DEC-SF: solid fraction after decanter, SCP-SF: solid fraction after screw press, P-POOR SF: solid fraction decanter and P stripping, DRY-SF: solid fraction after decanter and drying, COMP: compost, BCHR: biochar produced from P-POOR SF.
Table 6. Pearson correlation coefficients among parameters related to product characteristics and C and N mineralisation dynamics

|            | TOC | N\textsubscript{tot} | N\textsubscript{min} | C/N\textsubscript{tot} | C/N\textsubscript{org} | N\textsubscript{min}/N\textsubscript{tot} | N\textsubscript{org}/N\textsubscript{tot} | WSC | HWEC | CA | HC | %C\textsubscript{min} | %N\textsubscript{min} |
|------------|-----|-----------------------|-----------------------|------------------------|------------------------|------------------------------------------|------------------------------------------|-----|------|----|----|----------------------|----------------------|
| TOC        | 1   |                       |                       |                        |                        |                                          |                                          |     |      |    |    |                      |                      |
| N\textsubscript{tot} | -0.372 | 1                      |                       |                        |                        |                                          |                                          |     |      |    |    |                      |                      |
| N\textsubscript{min} | -0.353 | -0.062                 | 1                      |                        |                        |                                          |                                          |     |      |    |    |                      |                      |
| C/N\textsubscript{tot} | 0.273  | -0.372                | 0.005                 | 1                      |                        |                                          |                                          |     |      |    |    |                      |                      |
| C/N\textsubscript{org} | 0.310  | 0.441                 | -0.515                | 0.310                  | 1                      |                                          |                                          |     |      |    |    |                      |                      |
| N\textsubscript{min}/N\textsubscript{tot} | -0.390 | -0.482                | 0.855                 | -0.366                 | -0.404                | 1                                          |                                          |     |      |    |    |                      |                      |
| N\textsubscript{org}/N\textsubscript{tot} | 0.390  | 0.482                 | -1.000*               | -0.366                 | 0.404                 | -1.000**                                   |                                          |     |      |    |    |                      |                      |
| WSC        | -0.547 | -0.095                | 0.821*                | -0.434                 | -0.623                 | 0.617                                    | -0.617                                  | 1   |      |    |    |                      |                      |
| HWEC       | -0.496 | 0.234                 | 0.285                 | -0.849*                | -0.606                 | -0.071                                    | 0.071                                   | 0.500 |      |    |    |                      |                      |
| CA         | -0.340 | -0.358                | 0.762                 | 0.548                  | -0.475                 | 0.825*                                   | -0.825*                                 | 0.376 | -0.146 | 1 |    |                      |                      |
| HC         | 0.355  | 0.327                 | -0.794                | -0.497                 | 0.502                  | -0.824*                                   | 0.824*                                  | -0.436 | 0.112 | -0.996** | 1 |    |                      |                      |
| %C\textsubscript{min} | -0.397 | -0.234                | 0.825*                | 0.334                  | -0.572                 | 0.759                                    | -0.759                                  | 0.110 | -0.292 | 0.941** | -0.966** | 1 |    |                      |                      |
| %N\textsubscript{min} | -0.065 | 0.400                 | 0.331                 | -0.880**               | -0.104                 | 0.015                                    | -0.015                                  | 0.700 | 0.572 | -0.340 | 0.276 | -0.106 | 1 |                      |                      |

3 TOC (g kg\textsuperscript{-1}): total organic carbon, N\textsubscript{tot} (g kg\textsuperscript{-1}): total nitrogen, N\textsubscript{min} (g kg\textsuperscript{-1}): mineral nitrogen, N\textsubscript{org} (g kg\textsuperscript{-1}): organic nitrogen, N\textsubscript{min}/N\textsubscript{tot}: mineral nitrogen as % of added N after 112 days of incubation, %N\textsubscript{min}: N mineralised as % of added organic N after 112 days of incubation

4 C\textsubscript{A}: amount of mineralisable C, HC: humification coefficient (%), %C\textsubscript{min}: C mineralized as % of added C after 81 days of incubation

5 WSC (% of TOC): water soluble carbon, HWEC (% of TOC): hot water extractable carbon, N\textsubscript{org}: organic nitrogen, %C\textsubscript{min}: C mineralized as % of added C after 81 days of incubation
Fig. 1. Cumulative amount of C mineralised after addition of 9000 kg OC ha$^{-1}$ to soil during the 81-day incubation period at 10 °C (mean value ± standard deviation, n=3). Lines represent the curve-fitting result; symbols are experimental data. Different lower case letters indicate significant differences between C mineralisation means at day 81. DEC-SF, solid fraction after decanter; SCP-SF, solid fraction after screw press; P-POOR SF, solid fraction decanter and P stripping; DRY-SF, solid fraction after decanter and drying; COMP, compost; BCHR, biochar produced from P-POOR SF.
Fig. 2. Evolution of (a) ammonium-N (b) nitrate-N and (c) total mineral N expressed in mg kg$^{-1}$ in the unamended soil and soil amended with the tested products during 112-day incubations.
at 21.5 °C (mean value ± standard deviation, n=4; where absent, error bars fall within symbols). SC, soil control; DEC-SF, solid fraction after decanter; SCP-SF, solid fraction after screw press; P-POOR SF, solid fraction decanter and P stripping; DRY-SF, solid fraction after decanter and drying; COMP, compost; BCHR, biochar produced from P-POOR SF
Fig. 3. N mineralisation (% of organic N supplied (equation 4) from the tested products during 112 days of incubation) (mean value ± standard deviation, n=4; where absent, error bars fall within symbols). DEC-SF, solid fraction after decanter; SCP-SF, solid fraction after screw press; P-POOR SF, solid fraction decanter and P stripping; DRY-SF, solid fraction after decanter and drying; COMP, compost; BCHR, biochar produced from P-POOR SF