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Research article

Pig slurry acidification and separation techniques affect soil N and C turnover and N₂O emissions from solid, liquid and biochar fractions



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ABSTRACT

The combined effects of pig slurry acidification, subsequent separation techniques and biochar production from the solid fraction on N mineralisation and N_2O and CO_2 emissions in soil were investigated in an incubation experiment. Acidification of pig slurry increased N availability from the separated solid fractions in soil, but did not affect N_2O and CO_2 emissions. However acidification reduced soil N and C turnover from the liquid fraction. The use of more advanced separation techniques (flocculation and drainage > decanting centrifuge > screw press) increased N mineralisation from acidified solid fractions, but also increased N_2O and CO_2 emissions in soil amended with the liquid fraction. Finally, the biochar production from the solid fraction of pig slurry resulted in a very recalcitrant material, which reduced N and C mineralisation in soil compared to the raw solid fractions.

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1. Introduction

Livestock production is developing dramatically on a global scale, with a trend towards increasing intensification on large specialised production units to improve profitability (Steinfeld et al., 2006). As a consequence, a high geographical concentration of manure production may lead to water pollution and nutrient leaching, soil contamination by heavy metal and pathogens, and gaseous emissions of odours, ammonia (NH₃), methane (CH₄), nitrous oxide (N₂O) and carbon dioxide (CO₂) (Sleutel et al., 2006). Livestock production contributes around 18% to global greenhouse gas (GHG) emissions according to Steinfeld et al. (2006). However, animal manure also contains high amounts of nutrients and organic matter that are essential for crop production (Schröder, 2005) and therefore the use of animal manure as a fertiliser could improve soil fertility and replace significant amounts of mineral fertilisers.

Several methods of animal manure processing have been developed for various purposes, including improved ease of handling, reduced costs associated with transportation, mitigation of environmental emissions, bioenergy production and soil amelioration. Mechanical solid—liquid separation of slurry is a relatively low-cost technology, which mainly aims to improve

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manure management options. Various slurry separation technologies are commercially available, including screw presses, filter bands, decanting centrifuges, chemical flocculation or a combination of several of these (Hjorth et al., 2010). In general, these techniques produce two fractions, a solid fraction with a high dry matter, organic nitrogen (N) and phosphorus (P) content which can be easily transported, and a liquid fraction, poor in organic N and with a low dry matter content, but high ammonium-N and potassium (K) content (Fangueiro et al., 2012). However, the main physical and chemical characteristics of the solid and liquid fractions depend on the technology used for separation (Jorgensen and Jensen, 2009).

Acidification of slurry is effective at reducing NH₃ emissions during animal housing, manure storage and soil application (Kai et al., 2008), but previous studies have shown that incorporation of acidified slurry into the soil can affect microbial processes such as soil C and N dynamics (Sørensen and Eriksen, 2009). Fangueiro et al. (2010) have observed a delay in nitrification in soil amended with acidified pig slurry. In the same study they report that CO₂ evolution decreases after the application of acidified solid and liquid fraction of pig slurry compared with non-acidified fractions.

Pyrolysis of the solid fraction from slurry separation has also been proposed as a technology for utilising some of the energy content of the solid fraction while producing a soil amendment product that has a positive effect on soil fertility (*e.g.* Tsai et al., 2012; Christel et al., 2014). Pyrolysis is a process whereby a

biomass feedstock is heated to high temperatures in an oxygen-free atmosphere to produce a solid (biochar), a liquid (bio-liquid) and a gaseous fraction. The proportion of biochar, bio-liquid and gas produced is dependent on the heating rate, the reactor temperature and the residence time of the reactor (Singh et al., 2010). The biochar can be used as a fuel or alternatively applied to land as a low-grade fertiliser, as a soil amendment or for carbon sequestration (Lehmann and Joseph, 2009). Biochar has been shown to have potential for reducing nutrient leaching in soils (Laird et al., 2010), increasing nutrient availability for plants and enhancing the efficiency of fertilisers as well as serving as a slow-release P fertiliser (Christel et al., 2014). Biochar has also been shown to influence soil physico-chemical properties such as pH, porosity, bulk density and water-holding capacity (Glaser et al., 2002).

However, the fertiliser value of the end products and the environmental effect of the treated manure in soil will depend on the manure composition and management systems used. The objectives of this study were to evaluate the combined effects of pig slurry acidification, the subsequent separation technique and biochar production from the solid fraction on: i) N mineralisation and N_2O emissions in soil, ii) CO_2 emissions and C accumulation in soil and iii) the effect of soil water content on these processes. It was hypothesised that i) acidification leads to a higher content of available N, but reduced N and C turnover, ii) a more efficient separation technique (in terms of dry matter) results in greater differences in N and C turnover between separation fractions and iii) pyrolysis of the solid fraction into biochar leads to high recalcitrance of remaining N and C, and reduced N_2O emissions.

2. Materials and methods

2.1. Pig slurry fractions and soil samples

Pig slurries from finishing pigs were collected from two separate pig pens at the Pig Research Centre — Grønhøj Experimental Station, Denmark (56°38′ N, 9°18′ E) during spring 2012. Untreated pig slurry (NA, from pen 1) and pig slurry continuously acidified (A, from pen 2) to pH 5.5 (with sulfuric acid, H₂SO₄) in the animal house were separated using three different separation technologies: (i) a screw press (SP) (Börger BS50, Börger GmbH, Borken-Weseke, Germany), (ii) flocculation using polymers and drainage with a filter band separator (FD) (AL-2 model 812P, Hovborg, Denmark), and (iii) a decanter centrifuge (DC) (Pieralisi, Gruppo Pieralisi — MAIP S.p.A, Jesi (AN) Italy). Further details on the slurries and separation techniques are described in Sommer et al. (2015). The solid (SF) and liquid (LF) fractions from the different pig slurry acidification and separation techniques were kept frozen until further analysis and experiments.

Biochar was produced from the solid fraction of pig slurry. The slurry solid fraction was first air-dried (forced ventilation, 30 °C, until 13% gravimetric moisture content) and pelletised (at JS Trading ApS, Fredericia, Denmark). Later the pelleted manure solids were slowly pyrolysed in a self-constructed bench-periodic reactor of 1.5 L capacity at the University of Limerick, Ireland. Oxygen was evacuated from the reactor at low temperatures in the early heating phase (heating rate 20 °C min⁻¹). During pyrolysis, no air could enter the reactor, but pyrolysis gases could escape. The manure solid feedstock remained at a peak temperature of 400 °C for 1 h, resulting in six different chars (CF).

A sandy loam soil was collected from the CRUCIAL field trial in October 2013 at Taastrup, Denmark. The soil was sampled from a plot that has received NPK fertiliser annually since 2003, containing approximately 100 kg inorganic N ha $^{-1}$ y $^{-1}$ and $<\!10$ kg P ha $^{-1}$ y $^{-1}$. The crop rotation at the plot has included spring and winter cereals, oilseed rape and grass. The soil was collected from the top 20 cm of

the soil profile. In the laboratory, the soil was sieved to 4 mm and stored at 4° C before the start of the incubation experiment.

Soil and pig slurry fraction samples were dried to 105 °C to estimate moisture content (DM). Volatile solids (VS) were estimated in dry samples that were combusted in a muffle oven (550 °C). All the samples were analysed for pH at a 1:5 ratio DI water (w:w). Soil and pig slurry fractions were finely ground before determination of total C (TC), using an elemental analyser and IRMS (Sercon ANCA-GSL, 20-20, Crewe, UK) and total Kjeldahl nitrogen (TN) was determined using a Kjeltec 2011 instrument (Foss, Höganäs, Sweden) according to the procedures given by APHA (2005). The contents of ammonium (NH $^{\perp}_4$) and nitrate (NO $^{-}_3$) were analysed by flow injection analysis as described below. Initial physical and chemical properties of the different fractions of pig slurry and soils are shown in Table 1.

2.2. Potential mineralisable N

An anaerobic incubation assay was employed to estimate potential mineralisable N (PMN) using a modified methodology proposed by Lober and Reeder (1993). 5 g dry soil was amended with the different fractions of pig slurry in an amount equivalent to 5 mg total N g $^{-1}$ in a 25 ml glass Erlenmeyer flask. Unamended soils were incubated as controls. 25 ml of distilled water was added to each sample, and then half of the samples were incubated at 37 °C for seven days, with the other half immediately extracted by adding 25 ml of 2 M KCl. The mixes were shaken for 1 h and filtered through Whatman no. 44 filter paper, and the NH $^{\downarrow}_{4}$ content was then measured by flow injection analysis (FIAstar 5000 flow injection analyser (Foss Analytical, Denmark)). The PMN was calculated according to the equation proposed by Fangueiro et al. (2009):

$$\begin{split} \text{PMN} &= \left\{ \left(\left[\text{NH}_4^+ \right]_7 - \left[\text{NH}_4^+ \right]_0 \right)_s - \left(\left[\text{NH}_4^+ \right]_7 - \left[\text{NH}_4^+ \right]_0 \right)_c \right\} \\ &\times \left/ \text{N applied} \times 100 \end{split} \tag{1}$$

where ($[NH_4^+]_7 - [NH_4^+]_0$)_s is the difference in the NH_4^+ concentrations measured on day 7 and day 0 in the samples and ($[NH_4^+]_7 - [NH_4^+]_0$)_c is the difference in the NH_4^+ concentrations measured on day 7 and day 0 in the control soil.

2.3. Incubation experiment

For the aerobic incubation experiment, each incubation vessel (100 ml plastic pots) received 50 g (dry weight) of sieved soil (<4 mm) and distilled water was added to reach pF 2 (estimated using a sandbox soil water equilibration system). The soils were pre-incubated in the pots for 1 week at 15 °C in the dark. After the pre-incubation period, three replicates of soil received one of the pig slurry fractions at a rate equivalent to 400 kg N total ha⁻¹ $(154 \text{ mg N kg}^{-1})$. In soil samples treated with liquid fractions of pig slurry, the lids were removed during the last three days of preincubation to increase water loss. This was done to ensure that when liquid fractions were applied, the resulting soil water content was not greater than pF 2. Unamended samples were also incubated as a control. All the samples were repacked to reach a bulk density of 1.3 g cm⁻³. One set of samples was adjusted to a soil water potential of pF 2 (-10 kPa), corresponding to field capacity (24% water w/w and 60% WFPS), and another set received distilled water to reach pF 1 (-1 kPa), corresponding to near-saturation (38%) water (w/w) and 97% WFPS respectively). The incubation was carried out for 160 days at 15 °C in the dark. During this period the moisture content of the soils was monitored by weighing, and lost water was replaced every two weeks.

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