



# CO<sub>2</sub> emissions and mineral nitrogen dynamics following application to soil of undigested liquid cattle manure and digestates



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## ABSTRACT

Anaerobic digestion of animal manures, and separation of digestates into a liquid (LF) and a solid fraction (SF) affect manure N availability to crops. Digestates and LF potentially supply more readily available N to crops compared to undigested manures and SF, due to their higher ammonium content, lower C to organic N ratio and lower dry matter content. Studies on digestates decomposition in soil are still scarce compared to those on undigested manures. We therefore carried out an incubation experiment to measure CO<sub>2</sub> emissions and soil mineral N dynamics following addition to soil of: ammonium sulphate (AS); digested cattle slurry-maize mix (DSMM); LF and SF of DSMM; undigested cattle slurry (US). The incubation lasted 181 days and was conducted at 25 °C and constant soil water content. Mineralization of C from US (51% of manure C after 181 days) was higher than that of digested manures (32–34%). Availability of applied ammonium after 181 days was similar for AS, DSMM and LF (70–78% of added ammonium), and was higher than that of SF and US (8 and 46% of added ammonium, respectively). Physical-chemical differences among manures explained main differences in decomposition dynamics. Indeed, US and SF induced net N immobilization (9–16% of manure organic N at day 181) due to high C to organic N ratio, high cellulose and volatile fatty acids content. Conversely, DSMM and LF induced net N mineralization (≈ 30% of manure organic N) due to a low C to organic N ratio and cellulose content. The findings of this laboratory experiment confirm the availability of manure N previously measured in a field experiment, where the same manures were applied to the same soil for the fertilization of silage-maize.

## 1. Introduction

Animal manures are a suitable source of nitrogen (N) for crops because they provide readily available mineral N (short-term effect), while organic N mostly contributes to long-term and residual fertilizer effects (Gutser et al., 2005). Efficient use of manure N requires adopting techniques that maximize plant N uptake and limit losses of N into the atmosphere, and the contamination of surface and ground water. These techniques shall be based on a thorough understanding of factors and processes that maximize manure N efficiency.

The efficiency of manure N depends on manure physical-chemical properties, as well as application period and method (Webb et al., 2013). Variability of manure composition firstly originates from animal species and diet (Kyvsgaard et al., 2000; Powell et al., 2006; Sørensen and Fernández, 2003; Sørensen et al., 2003), and type of husbandry; in addition, other biotic or abiotic processes could further modify manure characteristics, and possibly their N efficiency (Gale et al., 2006; Morvan et al., 2006; Peters and Jensen, 2011). Among these processes, anaerobic digestion was reported to generally enhance the ammonium

(NH<sub>4</sub>-N) share of manures, to reduce and to stabilize manure organic matter, and to reduce manure C to N ratio, resulting in potentially higher N availability for crops from digestates compared to undigested manures (Möller and Müller, 2012; Webb et al., 2013). In addition, separation of manures into a liquid and a solid fraction further affects their N efficiency. Indeed, liquid slurries with low dry matter (DM) content, low C to N ratio, and with high N and NH<sub>4</sub>-N content usually have high N efficiency because they quickly infiltrate into the soil, thus reducing N losses into the atmosphere; during decomposition they also induce a positive net N mineralization (or at least a low net N immobilization), and provide high amounts of readily available N for crops (Delin et al., 2012; Sørensen and Fernández, 2003; Sørensen et al., 2003). Conversely, manures characterized by high DM content, large C to N ratio, and low NH<sub>4</sub>-N content, like the fibrous (solid) fraction of manures, supply less available N to crops, at least in the short-term (Cavalli et al., 2016a; Gutser et al., 2005; Peters and Jensen, 2011; Sørensen and Thomsen, 2005). However, the literature about N availability from digestates and their fractions is still scarce compared to that on undigested manures (Cavalli et al., 2016a; Möller and Müller,

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2012).

Usually manure N availability is studied with field trials, where decomposition occurs in the presence of crops and under local weather conditions. Another approach involves incubation experiments carried out in the laboratory. In such experiments, controlled constant and optimal soil temperature and water content are maintained, and thus detailed C and N dynamics can be measured, without the confounding effects of soil spatial heterogeneity, weather and crop N uptake (Delin et al., 2012). Incubation experiments clearly indicate that short-term net N immobilization usually occurs in the first weeks after manure addition to soil, while net N mineralization is often observed in the following weeks (Bechini and Marino, 2009; Morvan et al., 2006). However, the question arises whether manure decomposition and N availability measured under controlled conditions actually reflect those observed in the field. If this holds true, as suggested by some experiments (Delin et al., 2012; Gale et al., 2006; Sørensen and Fernández, 2003; Sørensen et al., 2003), further integration of results obtained under laboratory and field experiments could improve our predictions of manure N availability for crops.

We recently published the results of a three-year field experiment aimed at measuring maize N uptake dynamics and the efficiency of N supplied with undigested cattle slurry and digestates (Cavalli et al., 2016a). Here, we report the results of an incubation experiment conducted with the same soil and the same manures used in the field trial. The two objectives of the incubation experiment were the following: 1) to analyze C and N mineralization dynamics in relation to manure composition; 2) to verify if incubation experiments provide results similar to field experiments concerning crop N availability after manure application.

## 2. Materials and methods

### 2.1. Treatments and experimental set-up

In the incubation experiment the six treatments, arranged in a completely randomised design with four replications, were the following: 1) unfertilised control (CON); 2) mineral fertilizer control (ammonium sulphate, AS); 3) unseparated digestate obtained from a mix of cattle slurry and silage maize (DSMM); 4–5) the liquid (LF) and solid (SF) fractions of DSMM; 6) unseparated anaerobically stored cattle slurry (US).

The three digested manures were taken from a biogas plant where cattle slurry was co-digested with silage maize (about 30% on a fresh matter basis) and beet pulp or tomato peels (about 1% on a fresh matter basis). The LF and SF manures were mechanically obtained from DSMM after screw press separation. The US was collected from a second farm where the storage tank lay beneath the litter-free, gridded stable floor. The main characteristics of the four manures are summarised in Table 1.

The loam soil used in the experiment (Table 2) was sampled from the 0–30 cm profile of a field that received no organic fertilization during the decade preceding its collection in spring 2011 (Cavalli et al., 2016a). Prior to the start of the experiment, the soil was air-dried and sieved until passable through 2 mm mesh. Thereafter, it was re-moistened and pre-incubated at 25 °C for one week.

After that week, treatments were applied to experimental units (microcosms) consisting of pre-incubated soil (equal to 100 g dry weight) at a rate of 61 mg NH<sub>4</sub>-N kg<sup>-1</sup> of dry soil, except CON that received no N addition.

The incubation study was carried out in the dark at 25 °C and constant soil water content (WC<sub>-50kPa</sub>, Table 2) and lasted 181 days, during which measurements of CO<sub>2</sub> emissions, soil N and pH were taken on 11 dates: 1, 3, 7, 14, 21, 28, 35, 42, 70, 126, and 181 days after the addition of fertilizers to the soil. In addition, soil N and pH were measured at day 0, more precisely 2 h after fertilizer application. In order to allow destructive measurements on different experimental

**Table 1**  
Characteristics of the four manures used in the incubation experiment.

Variable/unit	Manure <sup>a</sup>			
	DSMM	LF	SF	US
Dry matter (DM)/g kg <sup>-1</sup>	65.1	47.9	256.5	82.3
Ash/g kg <sup>-1</sup>	16.9	12.5	35.3	14.2
pH	8.0	8.0	9.6	7.3
Total C (TC)/g C kg DM <sup>-1</sup>	395.8	363.6	439.8	436.4
Volatile fatty acids C (VFA C)/% TC	0.2	0.2	0.0	7.9
Soluble C/% TC	55.8	55.5	25.2	35.5
Hemicellulose C/% TC	0.0	0.0	8.5	13.2
Cellulose C/% TC	18.1	15.6	31.5	26.5
Lignin C/% TC	25.9	28.7	34.8	16.8
Total N (TKN)/g N kg DM <sup>-1</sup>	55.9	67.0	21.9	39.2
Ammonium-N (NH <sub>4</sub> -N)/% TKN	45.6	51.0	23.3	53.1
Soluble N/% TKN	36.2	31.9	37.2	32.1
Hemicellulose N/% TKN	0.0	0.0	0.0	1.1
Cellulose N/% TKN	2.4	1.4	6.5	3.2
Lignin N/% TKN	15.8	15.6	33.0	10.5
TC/organic N	13	11	26	24
Estimated partitioning coefficients of DSMM between LF and SF				
Dry matter/% DM DSMM	100	68	32	–
Total C/% TC DSMM	100	63	37	–
Total N/% TKN DSMM	100	86	14	–
Ammonium-N/% NH <sub>4</sub> -N DSMM	100	93	7	–

<sup>a</sup> DSMM, unseparated digestate from a mix of cattle slurry and maize; LF, liquid fraction of DSMM; SF, solid fraction of DSMM; US, anaerobically stored untreated cattle slurry.

**Table 2**  
Characteristics of the soil used in the incubation experiment.

Variable/unit	Value
Sand/g kg <sup>-1</sup>	469
Silt/g kg <sup>-1</sup>	394
Clay/g kg <sup>-1</sup>	137
Total C/g kg <sup>-1</sup>	8.4
Total N/g kg <sup>-1</sup>	1.0
Exchangeable K/mg kg <sup>-1</sup>	167
Exchangeable Mg/mg kg <sup>-1</sup>	242
Extractable P (Bray and Kurtz method)/mg kg <sup>-1</sup>	61
pH in water	5.8
Water content at –50 kPa (WC <sub>-50kPa</sub> )/g H <sub>2</sub> O kg <sup>-1</sup>	144
Bulk soil	
Quartz/g kg <sup>-1</sup>	370
K-Feldspar/g kg <sup>-1</sup>	80
Plagioclase/g kg <sup>-1</sup>	90
Amphibole/g kg <sup>-1</sup>	40
Chlorite/g kg <sup>-1</sup>	160
Mica/illite/g kg <sup>-1</sup>	140
Vermiculite/g kg <sup>-1</sup>	50
Kaolinite/g kg <sup>-1</sup>	70
Fraction ≤ 2 μm	
Quartz/g kg <sup>-1</sup>	50
Chlorite/g kg <sup>-1</sup>	110
Mica/illite/g kg <sup>-1</sup>	270
Vermiculite/g kg <sup>-1</sup>	240
Kaolinite/g kg <sup>-1</sup>	330

units on each sampling date (Thuriès et al., 2000), we prepared 288 experimental units (6 treatments × 12 sampling dates × 4 replicates) for which measurements were done only once.

### 2.2. Analysis of manures and soil

Before analysis and incubation, liquid manures were homogenised using an Ultra Turrax T-25 disperser (IKA Werke GmbH & Co. KG, Germany), while the solid fraction was hand-chopped. All manures

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