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(Article begins on next page)

UNIVERSITÀ DEGLI STUDI DI TORINO

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Gaseous emissions from the storage of untreated slurries and the fractions obtained after mechanical separation

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Abstract

38 A laboratory scale study was set up to investigate the ammonia (NH₃), nitrous oxide (N₂O), carbon 39 dioxide (CO_2) and methane (CH_4) emissions during storage of untreated pig and cattle slurry and the fractions (solid and liquid) obtained by mechanical separation. The solid and liquid fractions were obtained from the same untreated slurry by means of a lab scale mechanical separator.

42 The manures were stored for a period of 30 days in open vessels $(1500 \text{ cm}^3 \text{ capacity})$ in two 43 temperature controlled rooms which were kept at 5 ± 0.5 °C and 25 ± 0.2 °C. Gaseous emissions were determined using a dynamic chamber method and Infrared Photoacoustic Detection (IPD).

 Over the storage period, gaseous emissions from pig manures were significantly (P<0.05) higher 46 than those from cattle manures. N₂O fluxes of up to 232 mg m⁻² h⁻¹ were measured but from pig 47 solid fraction only. Between 40.8% (from pig liquid fraction stored at 25 °C) and 3.60% (from 48 untreated cattle slurry stored at 25° C) of the initial nitrogen content of the manures was lost as NH₃. Over the 30-days storage period, the predominant emission of carbon was in the $CO₂$ form. Total C-CH⁴ losses expressed as a percent of the carbon initially present in the volatile solids (C- VS) ranged from 0.60% to 12.8% for pig manures and from 0.23% to 1.56% for cattle manures. The 52 mechanical separation of cattle slurry increased by up to 30% the emissions of $CO₂$ equivalents to the atmosphere during the storage of the separated fractions if compared with the slurries. Results 54 indicated that NH₃, N₂O, CO₂ and CH₄ emissions were affected by the interaction between a number of variables, including storage temperature, chemical characteristics and type of manure. Results also indicated that mechanical separation does not reduce emissions but has the potential to increase the emissions of $CO₂$ equivalents to the atmosphere during the storage of the separated fractions.

Keywords: ammonia, greenhouse gas emissions, manure storage, slurry separation.

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1. Introduction

 A press zone was created fitting the blade to the disc by means of a horizontal bar and a spring. After filling the bowl with the untreated slurry, the blade is manually rotated using the crank and the slurry separated in the press zone. The liquid fraction runs through the disc's holes into the container while the solid fraction remains in the bowl and is collected afterwards. The total input of each untreated slurry (100 kg), as well as the amounts of solid (18.1 kg for cattle and 7.80 kg for pig) and liquid (81.9 kg for cattle and 92.2 kg for pig) recovered, were weighed and recorded. The 107 bulk density of the solid fractions (cattle and pig) was estimated to be 0.40 t m^{-3} .

108 Immediately after mechanical separation, samples of 1000 cm^3 of each of the investigated materials 109 were stored for a period of 30 days in open vessels $(1500 \text{ cm}^3 \text{ capacity}, 0.20 \text{ m} \text{ height}, 0.10 \text{ m} \text{ base})$ 110 diameter and 0.095 m top diameter) in two temperature-controlled rooms which were kept at 5 ± 0.5 111 and 25 ± 0.2 °C. During the test period observations of crust formation and reduction of volume were carried out. Four replicates of each product were tested so that for each type of untreated liquid manure (pig or cattle) a total of 12 vessels were used per climate room.

 The samples were analysed for pH, total solids (TS), volatile solids (VS), total Kjeldhal nitrogen (TKN), total ammoniacal nitrogen (TAN), and organic acids at the beginning and at the end of the investigation period. Initial and final slurry analyses are given in Table 1.

 Flux measurements were carried out using infrared photoacoustic detection (IPD) and the dynamic chamber method described by Berg et al. (2006). This method allows the comparison of the effect of different treatments on gaseous emissions during manure storage, under standardized experimental conditions. Different conditions, such as volume to surface ratio of the manure stores, in the lab and on farms may yield different gas fluxes. Therefore, this fluxes cannot be used for calculating fluxes from on farm storage facilities without further validation. Accordingly, the slurries were stored in open vessels using the dynamic chamber method set up only during measurements. Before starting the measurements each vessel was closed with an air-tight lid provided with two ports for air inlet and outlet (Fig. 2). Air inlet port was connected in an airtight

127 way with a flow meter and a compressor. The headspace (chamber) between the slurry surface and

128 the lid was then ventilated with compressed air to create an airflow through the dynamic chamber.

129 The air exchange inside the chambers was adjusted by the flow meters so that the air in the

130 headspace was always changed once per minute. Measurements were carried out four times a week 131 according to the following scheme:

132 1) lid closure at time $t = 0$ min;

- 133 2) between t=0 and t= 20 min the chamber was only ventilated to achieve steady conditions inside 134 the chamber;
- 135 3) t= 20 min beginning of the exhaust air sampling;

136 \neq 1 t = 36 min end of the exhaust air sampling and vessel opening.

 The gas concentrations in the inlet (atmospheric) and outlet (exhaust) air were analyzed by the INNOVA system 1312 Photoacoustic Multi-gas Monitor and Multipoint Sampler. The system 139 provided a real time analysis of 4 gases (NH_3 , N_2O , CH_4 and CO_2) plus water vapour. Measured 140 data were related to standard conditions (25 °C and 100 kPa) and stored in a database. Exhaust air leaving each chamber was sampled for 16 min. The instrument needed 2 min to analyze one sample, thus, 8 values were recorded on each measurement occasion. The first 3 values were discarded and the mean of the remaining 5 recorded values was considered representative for the measurement. 144 Gas fluxes F_j in mg m⁻² h⁻¹ were determined according to:

145
$$
F_j = Q \frac{(C_{ex,j} - C_{in,j})}{A}
$$
 (1)

146 where $C_{in,j}$ is the NH₃, N₂O, CH₄ or CO₂ concentration of air inlet the chamber in mg m⁻³; $C_{ex,j}$ is 147 the NH₃, N₂O, CH₄ or CO₂ concentration of air outlet the chamber in mg m⁻³; Q is the air flow rate 148 through the chamber in m³ h⁻¹; A is the area of emitting surface covered by the chamber in m². 149 Time evolution of the gas fluxes were approximated by spline approximation functions, and by 150 integration of such approximations using adaptive Simpson quadrature formulas (Bronshtein et al.,

151 2003) cumulative losses φ_j in mg vessel⁻¹ of each gas over the storage period (30 days) were 152 estimated.

153 Since measurements were carried out with 1000 cm^3 of all substrates respectively, in order to assess 154 the effect of mechanical separation on gaseous emissions, cumulative losses were corrected as 155 follows:

$$
156 \\
$$

$$
\Phi_j = \varphi_j \, \rho \tag{2}
$$

157 where Φ_j is the corrected cumulative loss of a specified gas in mg vessel⁻¹; ρ is a correction factor that takes into account the mass *Sc* (kg) of separated liquid and solid fractions obtained after separation of the untreated slurry *Mc* (kg), and the amount *Me* (kg) of untreated slurry, liquid and solid fractions used for emission trials:

$$
\rho = \frac{Sc}{Mc} \frac{1}{Me}
$$
 (3).

162 The ρ values used are summarized in Table 1.

 During storage trials, volume and surface area in contact with the air were the same for all tested materials. Nevertheless, in practical conditions slurries are stored within tanks and solid fraction in 165 heaps on platforms. The slurry NH₃ emitting area is the horizontal cross section of the storage tank, while for solid material it may be considered the surface area of a hypothetical frustum of cone 167 representing the shape of the heap. Considering an angle of repose of $1¹/2$: 1 (horizontal: vertical) (CNMP, 2002), the surface/volume ratio for the pig solid fraction was calculated to be 1.83 times as much as pig slurry and 2.44 times as much as cattle untreated slurry for the cattle solid fraction. 170 Thus, to be able to assess the effect of mechanical separation on $NH₃$ emissions, measured $NH₃$ emissions from pig solid fraction and cattle solid fraction were therefore multiplied by 1.83 and by 2.44 respectively, assuming that the emissions per unit area were homogeneous. The emitting surface area of the liquid fraction was assumed to be that of the untreated slurry.

174 To consider the effect of slurry mechanical separation on GHG emissions as a whole, the lC_i *l* were 175 converted into CO_2 equivalents by the Global Warming Potential (GWP) of the gases (NH₃: 3.1, 176 N₂O: 310, CH₄: 21) (IPCC, 1997).

 Significant differences in results and all possible interactions were investigated using the ANOVA procedure. Before analysis some of the parameters were log-transformed in order to fit a normal 180 distribution. For all the statistics, a significant level of $P = 0.05$ was applied. Assumption of equal variance of different groups was tested using Bartlett's test. All statistical analyses were performed with SPSS 12.0 for Windows.

3. Results

3.1 Slurry properties

 After mechanical separation the liquid fractions produced by mechanical separation showed significant reductions of TS and VS (Table 1). In contrast, the pH, TAN and TKN concentrations of the liquid fractions were very similar to those of the untreated slurries. As expected, the TS and VS content of the solid fractions were significantly greater than those for the untreated slurries. The TKN content of the pig solid fraction was almost double the TKN content of the pig untreated slurry, while the TKN content of the cattle solid fraction was approximately 1.4 times lower than the TKN content of the cattle untreated slurry. Analysis of the manures at the end of each experiment are given in Table 2 together with volume reductions over the storage period.

Water evaporation occurred in all the samples throughout the storage period. As expected,

196 evaporation was higher from materials stored at 25 \degree C than 5 \degree C and was responsible for volume

reductions up to 45% of the stored slurries and of the relative increase of TS and VS amounts.

198 Appreciable volume reduction (30.6%) occurred for cattle solid fraction stored at 25 °C, low

199 reductions were observed for solid materials stored at 5 °C while no volume reduction occurred for 200 pig solid fraction stored at 25° C.

201

202 3.2 Gaseous emissions

203 $3.2.1$ N₂O emissions

204 Over the storage period (30 days), no N₂O fluxes were measured from any material stored at 5 °C. 205 N₂O emissions from cattle and pig slurries stored at 25 \degree C occurred on a very few occasions only, 206 e.g. when the crust dried up, but the overall amounts were negligible. Very low concentrations, 207 sometimes slightly above the background concentration of N_2O , were also measured from the cattle 208 solid fraction. Appreciable N_2O fluxes were measured only from the pig solid fraction stored at 209 25 °C (Fig. 3). N₂O emissions started 8 days after the beginning of the storage period and reached a 210 peak of 232 mg m⁻² h⁻¹ at day 21. After that peak, the N₂O flux rapidly decreased to reach values 211 close to zero at day 25. The total nitrogen loss via the N₂O pathway accounted for the 4.71% of the 212 initial total Kjeldahl nitrogen (TKN) content of the solid manure (Table 3).

213

214 $3.2.2$ NH₃ emissions

215 Total losses of NH₃-N, expressed as a percentage of the initial TKN, (Table 3) ranged from 3.60% 216 (from untreated cattle slurry stored at 25 °C) to 40.8% (from pig liquid fraction stored at 25 °C). 217 Under the same storage conditions, there were no significant $(P>0.05)$ differences between NH₃ 218 fluxes from untreated pig slurry and from its liquid fraction (Fig. 4). In contrast, NH_3 fluxes from 219 untreated cattle slurry were significantly less than from its liquid fraction. $NH₃$ emissions were 220 higher at 25 °C than at 5 °C storage conditions, except when considering the pig solid fraction. 221 From this, significantly (P<0.05) higher NH₃ fluxes at 25 °C than at 5 °C were measured only at the 222 beginning of the storage period, while, after day 8 NH₃ fluxes were significantly lower at 25 $^{\circ}$ C 223 than at 5° C storage conditions.

225 $3.2.3 \text{ CO}_2$ and CH₄ emissions

226 During pig and cattle manure storage, the predominant emission of carbon was as $CO₂$ (Fig. 5 and 227 Table 3). Total losses of $C-CO₂$ expressed as a percentage of the carbon initially present in the 228 volatile solids (C-VS) of the manures ranged from 7.76% (from cattle untreated slurry stored at 229 5 °C) to 33.4% (from pig liquid fraction stored at 25 °C). CO₂ losses were always higher at 25 °C 230 and lower at 5° C storage conditions.

231 Under the same storage conditions, CH₄ fluxes from the untreated slurries and from their liquid 232 fractions were not different (P>0.05) (Fig. 6), but total C-CH₄ losses over the storage period, 233 expressed as a fraction of initial C-VS content, were significantly (P<0.05) higher from liquid 234 fractions than from untreated slurries (Table 3). Under all storage conditions, total C-CH₄ losses 235 were higher from pig than from cattle slurries. C-CH₄ losses ranged from 0.60% to 12.8% for pig 236 manures and from 0.23% to 2.19% of the initial C-VS content for cattle manures. C-CH₄ losses 237 from pig manures were always consistently higher at 25 \degree C than those at 5 \degree C. However, the 238 emissions of CH⁴ were only observed to be significantly (P<0.05) higher from cattle slurries stored 239 at 25 °C than at 5 °C during the first 3 days of storage. Thereafter, the emissions were lower from 240 cattle slurries stored at 25 °C than at 5 °C. C-CH₄ losses from cattle and pig solid fractions were 241 always significantly lower at 25 $\mathrm{^{\circ}C}$ than at 5 $\mathrm{^{\circ}C}$ storage.

242

243 3.2.4 Effect of slurry solid-liquid separation on gaseous emissions

244 Figure 7 shows the NH₃, CO₂ and CH₄ losses based on the corrected cumulative losses *lC_il* and 245 expressed as a percentage of the *lCjl* calculated for the untreated slurries (pig and cattle). It can be 246 seen that for pig slurry the combined emissions measured from the storage of the liquid and solid 247 fractions resulted in reduced NH³ losses compared to the storage of the untreated pig slurry. 248 However for cattle slurry, the combined NH₃ losses measured from the storage of the liquid and 249 solid fractions were 7% and 45% higher than those measured from the untreated cattle slurry stored 250 at 25 C and 5 °C respectively.

251 The emissions from the storage of the two fractions increased the $CO₂$ emissions from 8% (when 252 compared to untreated pig slurry stored at 5° C) to 104% (when compared to untreated cattle slurry 253 stored at 25 °C). During the storage of both liquid and solid fractions, CH₄ losses were 3% higher 254 for pig untreated slurry stored at 25 \degree C and 4% higher for cattle untreated slurry stored at 5 \degree C. 255 Instead, solid-liquid separation reduced CH₄ emissions by 8% for pig untreated slurry stored at 5 $^{\circ}$ C 256 and by 9% for cattle untreated slurry stored at 25 $^{\circ}$ C. 257 Combining the emissions of CO_2 equivalents from both liquid and solid fractions (Fig. 8), they

258 resulted in increased greenhouse gases emissions compared to the storage of the untreated slurries, 259 except when considering the pig untreated slurry stored at 5 °C. In this latter case the mechanical 260 separation had no effect in combined emissions of $CO₂$ equivalents.

261

262 **4. Discussion**

263 The small N_2O fluxes from cattle and pig slurries storage can be explained by the absence of crust 264 during most of the storage period. N₂O may be emitted during storage of manure either as a by- product of incomplete ammonium oxidation or as a by-product of incomplete denitrification (Oenema et al., 2001). The condition in liquid manure is strictly anaerobic, and neither process 267 occurs. In contrast, authors (e.g. Sommer et al., 2000; Berg et al., 2006) found N_2O production from stored slurries, but only when a dry crust formed on the surface. These emissions occurred since surface crust may contain a mosaic of anaerobic and aerobic micro–sites which are favourable for N₂O production. Dry conditions, together with air filled porosity, may explain the occurrence of N₂O production from pig solid fraction samples. On the other hand, separation of cattle slurry produced a fibrous solid fraction with a compacted structure, this might have created an 273 environment with an oxygen concentration unsuitable for $N₂O$ production. Results confirm a positive relationship between ammonia emission and temperature found by other studies (e.g. Ni, 1999; Dewes, 1996), but not in the case of the pig solid fraction. Total losses from

276 the pig solid fraction were 7.12 and 5.57% of the initial TKN content at 5 and at 25 \degree C storage

conditions respectively. This may be explained by ammonium oxidation by nitrifying micro-

278 organisms with N₂O production. NH₃ emission from manure decreased, therefore, as a greater proportion of the NH4-N was oxidized by nitrifying micro-organisms.

 CH⁴ losses from pig slurries significantly increased with temperature (Table 3). This is confirmed by results of other studies (e.g. Husted, 1994), in which an increase of the CH⁴ production with air 282 storage temperature was also found. The lowest CH₄ emissions from cattle slurries stored at 25 °C 283 with respect to those obtained at 5° C were probably caused by the high water losses from the slurries over time. As a consequence, a relative increase in concentration of constituents, such as NH³ and organic acids, which could be inhibitory to the methanogens (Kalle and Menon, 1984; 286 Angelidaki et al., 1999), occurred. This was proved by the CH_4 fluxes from both untreated cattle slurry and liquid fraction stored at 25 °C which were observed to be high at the beginning of the storage and to gradually decrease over time (Fig. 6). Additionally, Table 2 shows for cattle slurries a relative increase in concentration of organic acids, which can be inhibitory to the methanogens, at the end of the storage compared to the beginning of the storage (Table 1). The combined effect of temperature and slurry concentration had no effect on CH⁴ losses from pig slurries, probably due to a more active methanogens population. According to Massé et al. (2003), manure from different animals probably contains different species of anaerobic bacteria, which may be better adapted or acclimatized to inhibitive components such as organic acids.

295 CH₄ emission reductions from the cattle and pig solid fractions stored at 25 °C could also be explained by the high water losses over time. From the beginning of the storage period, when solid fractions started to dry, air entered the pores previously filled with water. The high water losses 298 combined with low volume (1000 cm^3) of the samples in storage, resulted in a substantial reduction 299 of the anaerobic zone inside the cattle and pig solid fractions stored at 25° C. Methanogenesis is inhibited by low moisture content and oxygen availability in the manure. Furthermore, most studies show CH⁴ emission reductions due to the methane oxidation during the passage through the porous surface layers of manure heaps (Olesen, 2005). Biological CH⁴ oxidation is a process whereby

 methane is oxidised to carbon dioxide and water by methane oxidizing bacteria (methanotrophs) under aerobic conditions (Cicerone and Oremland, 1988; Kotsyurbenco, 2005). The substantial reduction of the anaerobic zone over time and the biological CH⁴ oxidation in the outer layers could 306 be the causes for lower CH₄ emissions from pig and cattle solid fractions stored at 25 °C.

5. Conclusion

 Results from this study show that emissions of NH³ and GHGs during the storage of animal 310 manures are influenced by different factors. Since emissions of NO_2 , NH_3 , CO_2 and CH_4 are affected by microbiological, chemical, and physical processes, complex interactions exist. This requires a whole system approach when pollutant abatement technologies have to be adopted. Evidence from the present study suggests that mechanical separation of cattle and pig slurries does 314 not reduce emissions but has the potential to increase the emissions of $CO₂$ equivalents to the atmosphere during the storage of the separated fractions by up to 30% if compared with the untreated slurries. The emission potential of pig slurry was up to 3 times higher than that of cattle slurry. It was determined under the laboratory conditions and assumptions as described. Thus, to confirm these results, experiments should be made under on-farm storage conditions.

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382 **Figure legends**

- 383 Fig. 1. Main components of the lab-scale mechanical slurry separator.
- 384 Fig. 2. Schematic diagram of the sampling system.
- 385 Fig. 3. Nitrous oxide (N₂O) fluxes from pig solid fraction stored at 25 °C. Error bars indicate standard error (number of
- 386 observations $= 4$).
- 387 Fig. 4. Emission fluxes of ammonia (NH₃) during storage of the tested materials at 5 °C and 25 °C. Error bars indicate
- 388 standard error (number of observations $= 4$).
- 389 Fig. 5. Emission fluxes of carbon dioxide (CO_2) during storage of the tested materials at 5 °C and 25 °C. Error bars
- 390 indicate standard error (number of observations = 4).
- 391 Fig. 6. Emission fluxes of methane (CH₄) during storage of the tested materials at 5 °C and 25 °C. Error bars indicate
- 392 standard error (number of observations $= 4$).
- 393 Fig. 7. Corrected cumulative loss Φ_j of NH₃, CO₂ and CH₄ of the investigated samples in relation to the Φ_j from
- 394 untreated slurries (pig and cattle) samples.
- 395 Fig. 8. Climatic warm potential of untreated slurry (pig and cattle), liquid and solid fractions expressed as $CO₂$
- 396 equivalents calculated from the cumulated emissions of the single trace gases.
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407 Fig. 3.

Fig. 4.

Fig. 5.

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Fig. 6.

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423 Fig. 7.

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Table legends

- Table 1. Main composition of the manures at the beginning of each experiment
- 438 (n=2) and the correction factors ρ used for Φ_j calculation.
- Table 2. Main composition of the manures at the end of each experiment (n=4) and volume reductions over the storage
- period.
- Table 3. Mean gaseous losses from each of the tested material occurred at the different experimental conditions after 30
- d of storage.
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448 Table 1.

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462 Table 3.

463 Error standards are given in parentheses (n=4). Means with unequal letters in each column are significantly different (P<0.05).

 $464 \text{ ND} = \text{not detected}$

465