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(Article begins on next page)

1 **FERTILIZER VALUE AND GREENHOUSE GAS EMISSIONS FROM SOLID FRACTION**
2 **PIG SLURRY COMPOST PELLETS**

3

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18

19 **ABSTRACT**

20 Conversion of pig slurry to pellets is a desirable fertilizer option for farmers who want to
21 mitigate environmental pollution from slurry accumulation. The goals of the current
22 investigation were to determine the fertilizer properties of pig slurry solid fraction (SF) pellets
23 and to assess its potential to enhance soil properties in order to reduce ammonia (NH₃)
24 volatilization and greenhouse gas (GHG) emissions. Various parameters influence SF-
25 based pellet fertilizer effectiveness: bulking agent use during composting, pellet diameter
26 sizing and soil application type (superficially or incorporated into the soil). Two composts
27 from the same pig slurry SF obtained from a screw press separator were prepared: pig SF
28 compost without a bulking agent (SSFC) and pig SF compost with wood chips as the bulking
29 agent (wood chip compost (WCC)). For each compost type, pellets of two different diameters
30 (6 and 8 mm) were produced. A mesocosm experiment, conducted with maize plants, was
31 used to test the fertilizer value of the considered pellets. In total, three compost fertilizers –
32 SSFC, WCC and nitrogen: phosphorus: potassium mineral fertilizer 15 : 15 : 15, plus one
33 unfertilized control treatment – were applied at the same N rate (equivalent to 200 kg/ha)
34 using two different methods (surface and soil incorporation). After 65 days, above-ground
35 biomass, roots and soil samples were collected and analysed. Subsequently, a second
36 mesocosm study was undertaken to measure NH₃ and GHG emissions released from pellet
37 fertilization. Ammonia volatilization was determined immediately after pellet application,
38 while carbon dioxide (CO₂), methane (CH₄) and nitrous oxide (N₂O) emissions were
39 monitored for 57 days. Study results indicated that both pellet types were effective slow-
40 release fertilizers for maize. Additionally, three actions seemed to make the nutrients
41 contained in pig SF compost pellets more available to plants: addition of a bulking agent
42 before com- posting, use of small diameter pellets and soil incorporation of the fertilizer.

43

44 **Key words:** composting; pelletizing; nutrient; NH₃; N₂O; CO₂; CH₄; maize.

45

46 INTRODUCTION

47 In several European countries, intensive pig produc- tion systems produce high quantities
48 of organic waste in limited and specific geographic areas. In Italy, the 6th Italian National
49 Census of Agriculture (ISTAT 2012) indicates that the regions of Piedmont, Lombardy and
50 Emilia-Romagna account for 90% of all pig breeding in the country (ISTAT 2012). In both
51 Europe and Italy, slurry storage for subsequent land application is the predominant manure
52 management practice, probably due to its simplicity, low cost and potential to reduce the
53 total cost of crop production as a chemical fertilizer replacement (Kunz et al. 2009).
54 However, the technique carries several environmental pollution risks: ammonia (NH₃) and
55 greenhouse gas (GHG) emissions into the atmosphere, nitrate (NO₃⁻) leaching into
56 groundwater and phosphorous (P) runoff into surface waters (Salazar et al. 2005; Rao et al.
57 2007; Troy et al. 2013; Zhu et al. 2014; Vazquez et al. 2015). Consequently, the European
58 Union and local authorities enforce regulations on application timings, distribution volumes
59 and proper techniques to manage the potential environment fallout of high volumes of pig
60 excreta generated in areas of its member countries (Berruto et al. 2013). At times, these
61 rules have unintended consequences, as does the Nitrates Directive (EEC 1991) that
62 restricts the animal manure nitrogen (N) application rate to 170 kg N/ha/year within defined
63 Nitrate Vulnerable Zones. In this case, the mandate fails to permit manure disposal in many
64 intensive livestock regions where cultivation occurs near farm facilities, increas- ing costs
65 for storage and transportation.

66 Several techniques have been developed to better manage livestock slurries (Jørgensen &
67 Jensen 2009). The separation of solid and liquid fractions (LFs) simplifies handling by
68 decreasing its volume. The LF, which is rich in soluble N (Fangueiro et al. 2012), is generally
69 applied in areas adjacent to the farm, while the solid fraction (SF), rich in nutrients and
70 organic matter (OM) (Fangueiro et al. 2012) and containing less water, can be applied to

71 land at greater distances. According to recent investigations (unpublished data), the SF can
72 be transported economically to fields up to 25 km from the livestock farm.

73 A promising approach to increase the benefits of pig slurry SF, as well as to create a
74 potential new market for pig slurry-derived fertilizer, is to pelletize it. The densification
75 process that occurs after composting increases the bulk density of SF from <500 to >1000
76 kg/m³ (Pampuro et al. 2013), which reduces transport, handling and storage costs (Kaliyan
77 & Vance Morey 2009). Furthermore, Alemi et al. (2010) and Romano et al. (2014) showed
78 that pelletizing homogenizes and further concentrates SF nutrients, thereby improving its
79 fertilizing and amending actions.

80 However, the high moisture content (75–80%) of fresh SF does not make it suitable for
81 pelletizing. In previous studies (Pampuro et al. 2014, 2016), turning windrow composting
82 has been revealed as a simple and cheap method to reduce the moisture content of SF. As
83 a consequence of the heat generated by composting, after only 72 days moisture can be
84 lowered by 40%, hence the material is suitable for pelletizing.

85 For optimizing the composting, a bulking agent is added to SF. This makes it possible to
86 adjust substrate properties such as air space, moisture content, carbon-to-nitrogen ratio
87 (C/N), particle density, pH and mechanical structure, positively affecting the decomposition
88 rate and, therefore, development of the temperature (Bernal et al. 2009). Lignocellulosic
89 agricultural and forestry by-products are typical bulking agents when composting N-rich
90 wastes such as animal manures (Bernal et al. 2009). Their low moisture and high C/N ratios
91 can improve the benefits of animal manures (Nolan et al. 2011). The most commonly used
92 materials are cereal straw, cotton waste and wood by-products (Ros et al. 2006; Bernal et
93 al. 2009; Nolan et al. 2011; Santos et al. 2016).

94 The current work aimed to determine the fertilizer properties, as well as the potential benefit
95 to improve soil properties and to reduce NH₃ volatilization and GHG emissions of pig slurry
96 SF pellets. Different techniques for SF-based pellet fertilizer production, including addition

97 of a bulking agent for composting, preparation of different pellet sizes and use of different
98 soil application methods, were investigated and tested within two separate mesocosm
99 experiments to control environmental conditions.

100 Several hypotheses have been formed: (1) compost derived from pig slurry SF can have a
101 significant short-term benefit as a fertilizer (not as an amendment only); (2) fertilizer
102 properties of SF-based pellets are not com- promised by the addition of a bulking agent for
103 com- posting; (3) reducing pellet diameter increases the availability of nitrogen: phosphorus:
104 potassium (NPK), and NH₃ volatilization and GHG emissions simultaneously; (4) soil-
105 incorporated pellets, as opposed to those applied superficially, reduce NH₃ volatilization and
106 GHG emissions while increasing nutrient availability.

107

108 **MATERIALS AND METHODS**

109 **Pellet preparation and characterization**

110

111 Two different composts were produced from the same SF obtained from a screw press
112 separator. The pig SF compost (SSFC) was obtained by composting 6000 kg of pig SF,
113 while the wood chip compost (WCC) resulted after composting 8000kg of the same pig SF
114 with 2400 kg of wood chips processed from urban garden pruning residues. During WCC
115 windrow preparation, materials were mixed thoroughly to achieve a theoretical C/N ratio
116 equal to 30 (Bishop & Godfrey 1983), so as to optimize compost- ing performance (Bernal
117 et al. 2009). After the set-up, windrows were placed on a concrete floor and the process was
118 monitored for 130 days. Each set consisted of two thermocouples placed at depths of 0·2 m
119 (T1) and 0·6 m (T2) from the windrow surface. Daily air temperatures were monitored and
120 recorded (Fig. 1). During the experimental period, windrows were turned six times (on days
121 7, 16, 28, 35, 50 and 71).

122 The two composts were pelletized to two different diameters ([Ø] 6 and 8 mm) by a
123 mechanical pelletizer (CLM200E, La Meccanica Srl, Padua, Italy).
124 A number of analyses were performed to characterize the four pellet types (two diameters
125 of two compost types): pH, moisture content, dry matter content (DM), total organic carbon
126 (TOC), total nitrogen (TN), ammonium nitrogen ($\text{NH}_4^+\text{-N}$), nitric nitrogen ($\text{NO}_3^-\text{-N}$), C/N, OM,
127 cation exchange capacity (CEC), total phosphorous (expressed as P_2O_5) and total
128 potassium (expressed as K_2O). The pH value was determined in a water-soluble extract 1 :
129 10 (w/w) using a Hanna HI 9026 portable pH meter fitted with a glass electrode combined
130 with a thermal automatic compensation system. Dry matter was calculated after drying at
131 $105\text{ }^\circ\text{C}$ for 12 h and OM content by loss on ignition at $430\text{ }^\circ\text{C}$ for 24 h (Navarro et al. 1993).
132 Samples for TOC analysis were prepared by drying the samples at $105\text{ }^\circ\text{C}$ for 24 h, followed
133 by treatment with sulphuric acid to eliminate any inorganic C, with subsequent analysis on
134 an elemental analyser (Carlo Erba Instruments). Total N and $\text{NH}_4^+\text{-N}$ were determined using
135 the Kjeldahl standard method. Nitric-N was determined by ion chromatography in a 1 : 20
136 (w/v) water extract (Garcia-Gomez et al. 2002); CEC was determined by sodium chloride
137 adsorption followed by the potassium nitrate displacement method (Silber et al. 2010). After
138 $\text{HNO}_3/\text{HClO}_4$ digestion, P_2O_5 was analysed by colourimetry and K_2O by flame photom-
139 etry (Garcia-Gomez et al. 2002). Table 1 reports the main chemical characteristics (mean
140 value of three replicates) for the pellets investigated.

141

142 **Fertilizer value experiment**

143 A mesocosm experiment was set up in a controlled environment ($22\text{ }^\circ\text{C}$) glasshouse to test
144 the fertilizer value of the different pig SF-based pellets in a randomized complete block
145 design with four replicates. The experiment included a total of ten treatments:

146 (1) SSFC Ø 6 mm superficially distributed [SSFC 6 SUP]; (2) SSFC Ø 8mm superficially
147 distributed [SSFC 8 SUP]; (3) SSFC Ø 6 mm mixed with the soil [SSFC 6 MIX]; (4) SSFC Ø

148 8 mm mixed with the soil [SSFC 8 MIX]; (5) WCC Ø 6 mm superficially distributed [WCC 6
149 SUP]; (6) WCC Ø 8 mm superficially distributed [WCC 8 SUP]; (7) WCC Ø 6 mm mixed with
150 the soil [WCC 6 MIX]; (8) WCC Ø 8 mm mixed with the soil [WCC 8 MIX]; (9) Conventional
151 mineral fertilization with NPK fertilizer (15–15–15) [NPK]; (10) unfertilized Control [CON].
152 Each experimental unit consisted of a plastic mesocosm pot (volume = 3.015 litre, diameter
153 = 160 mm, height = 150 mm) with small holes in the bottom for excess water drainage
154 containing clay–silty soil collected from the top 20 cm of the CEBAS–CSIC experi- mental
155 fields located in Santomera, Murcia Region (Spain). The soil was air-dried for 5–6 days and
156 sieved to <5 mm for the mesocosm experiment. For the soil characterization analyses
157 described above, soil was further sieved to <2 mm; results are reported in Table 2.
158 Each mesocosm was uniformly packed with 3 l of soil at a bulk density of 1350kg/m³ (Wu et
159 al. 2011). Initially, all pots were moistened with deio- nized water to attain a 60% water-filled
160 pore space (WFPS). The water added to each mesocosm was calculated to supply 70% of
161 the water holding capacity (WHC), which corresponded to 670ml per pot. Thereafter, soil
162 water content was adjusted via a drip irrigation system (4 litre/min for 10 min) every 2–5
163 days as required for the crop. Mesocosms were fertilized manually (with SSFC or WCC or
164 NPK mineral fertilizer) at a consistent N application rate (equivalent to 200 kg/ha).
165 Depending on pellet composition, P and K were supplied as follows to the soil: 240 kg
166 P₂O₅/ha and 60 kg K₂O/ha for SSFC; 255 kg P₂O₅/ha and 110 kg K₂O/ha for WCC; and 200
167 kg P₂O₅/ha and 200 kg K₂O/ha for NPK fertilizer. Maize (*Zea mays* L.) FAO 500 seeds were
168 then sown into the mesocosm pots at a density of two plants per pot. Plants grew for 65
169 days.
170 At the end of the trial, the above-ground biomass was harvested, roots were separated from
171 the soil and the soil was sampled. After washing both the above- and below-ground biomass
172 with tap and dis- tilled water (two times each), all were dried at 60 °C for 72 h and sub-
173 samples were milled to 0.5 mm for analysis and moisture content determination.

174 Soil pore water was sampled three times during the experiment (days 30, 60, 65) in the MIX,
175 NPK fertilizer and unfertilized control treatments using FLEX-type 'Rhizon' soil pore water
176 samplers (Rhizosphere Research Products, The Netherlands) inserted at the surface of
177 each pot at approximately 45°. Soils were wetted to saturation (100% of their WHC) with
178 deionized water 24 h prior to each pore water extraction to ensure soil solution equilibrium.
179 Nitrogen concentration was assessed by automatic microanalysis. After HNO₃-H₂O₂
180 microwave-assistant digestion, P composition of the aerial parts was determined by
181 colourimetry (Kitson & Mellon 1944) and K by flame photometry. Soil samples were analysed
182 for nitrate (NO₃) by ion chromatography in a 1 : 20 (w/v) water extract, while electrical
183 conductivity (EC) and pH were evaluated in a water-soluble extract 1 : 10 (w/v). An automatic
184 liquid sample analyser (TOC- V CSN + TNM-1 Analyser, Shimadzu, Tokyo, Japan) was
185 used to measure soluble N in pore water. All chemical determinations were performed in
186 duplicate.

187 Plant N utilization efficiency was calculated on the basis of the apparent recovery fraction
188 (ARF) approach (Gunnarsson et al. 2010), according to the following equation:

189

$$190 \text{ ARF} = (\text{N uptake treatment} - \text{N uptake control}) / \text{TN added}$$

191 in which N uptake treatment is the total N uptake (mg/pot) of a fertilizer treatment at
192 harvesting, N uptake control is the total N uptake (mg/pot) of the unfertilized control and TN
193 added is the total N added to each pot (mg/pot). A similar calculation was done for P, but
194 without subtracting P uptake of the control (Syers et al. 2008).

195

196 **Ammonia and GHG experiment**

197 A second mesocosm experiment, also of a randomized complete block design with four
198 replicates, was set up to measure NH₃ volatilization and GHG emissions. Nine of the ten

199 treatments described for the first experiment were included in this investigation; the 'NPK
200 treatment' was omitted.

201 The experiment was carried out in glass jars (3.2 litre capacity). To mimic the plough layer
202 (0– 30 cm) of the soil, all jars were filled with 1.5 kg of the same soil used in the first
203 mesocosm experiment; they were also moistened with deionized water to reach 60% of
204 WFPS (Subedi et al. 2013). Next, the soil was brought back to field density (1.35 g/cm^3 ; Wu
205 et al. 2011), at which the headspace volume equalled 2000 cm^3 . The jars were then pre-
206 incubated at 20 °C until the initial CO_2 flux from soil re-wetting had subsided (10 days). After
207 pre-incubation, jars were manually fertilized with either SSFC or WCC pellets with the same
208 nutrient amounts as described in the first experiment. Thereafter, all jars were main- tained
209 in a climate-controlled room at a constant 25 °C and air humidity of about 55%. The soil
210 moisture content of each jar was maintained at 60% WFPS for 57 days via gravimetric
211 adjustment every 2–3 days as required. No gas measurement was taken <12 h after an
212 adjustment.

213 Ammonia volatilization was measured for 48 h following pellet application at 20 °C and at
214 an air-flow rate of 2litre/min (Subedi et al. 2013) with a dynamic chamber system coupled
215 with a photoacoustic trace gas analyser (PTGA, INNOVA 1412, LumaSense Tech).

216 Emissions of the main GHG produced from agricultural soils (i.e., CO_2 , CH_4 and N_2O) were
217 measured from the jars three times weekly for the first 2 weeks after fertilization, then twice
218 weekly for the following 3 weeks and once weekly for the last 4 weeks, for a total of 16 times
219 during the 57-day period. Greenhouse gas fluxes were measured for each sealed jar using
220 a gas-tight polyethylene lid equipped with two Teflon tubes (each 5 cm long) punctured by
221 several small holes (0.5 mm diameter) to sample air from the entire headspace volume.
222 Thirty millilitres of air was withdrawn by plastic syringe from the jar headspace at 0, 9 and 18
223 min after jar closure. All samples were stored in airtight glass vials (12 ml Exetainer[®] vials)
224 and analysed for CO_2 , CH_4 and N_2O concentrations within 24 h by gas chromatography

225 (Agilent 7890). The gas chromatograph (GC) was equipped with thermal conductivity, flame
226 ionization and electron capture detectors for determination of CO₂, CH₄ and N₂O
227 concentrations, respectively. For each jar closure, concentrations of the three GHG were
228 plotted over time and fluxes were calculated with a linear or polynomial model, depending
229 on their specific accumulation pattern (Subedi et al. 2016). Cumulative emissions were
230 estimated assuming a linear change in fluxes between adjacent sampling points.
231 Total gaseous losses were expressed in CO₂-eq using conversion factors of 1, 28, 265 and
232 2.65 for CO₂, CH₄, N₂O and NH₃ (IPCC 2013), respectively.

233

234 **Statistical analyses**

235 One-way analysis of variance (ANOVA) was performed to evaluate all investigated variables
236 concern- ing plant, root, soil pore water, soil and cumulative NH₃ and GHG emissions. A
237 Kolmogorov–Smirnov test was used to test normality of distribution; homo- scedasticity was
238 verified with Levene’s test. For each variable, if treatment effect was statistically significant,
239 the ANOVA was followed by the planned contrasts test. Nine contrasts were planned; first,
240 the unfertilized control against all the fertilized treatments (all pellets + NPK); then NPK
241 against all pellet-fertilized treatments (SSFC + WCC); subsequently, SSFC pellets against
242 WCC pellets; afterwards, within each type of pellet (both SSFC and WCC) 6 mm diameter
243 against 8 mm diameter; finally, (within each type of pellet and each diameter) surface
244 application against soil mixed application. For apparent recovery (AR), only eight contrasts
245 were realized, excluding the unfertilized control.

246 Statistical analyses were performed by SPSS soft- ware (IBM SPSS Statistics for Windows,
247 Version 21.0. Armonk, NY: IBM Corp.).

248

249 **RESULTS**

250 **Maize biomass and nutrient concentrations**

251 All plants in all treatments appeared healthy through- out the growing period and did not
252 show any sign of nutrient deficiency or toxicity at any time. Fertilizer treatments significantly
253 affected above-ground yield ($P \leq 0.010$) and NPK concentrations ($P < 0.001$), as well as root
254 production ($P < 0.001$) and N concentration ($P < 0.001$) (Table 3).

255 Table 4 shows that after 65 days all fertilized treatments produced significantly greater yields
256 ($P < 0.001$) and NPK concentrations ($P < 0.001$) relative to the unfertilized control, while no
257 other difference was sig- nificant for maize yield. Pellet-fertilized maize exhibited lower N
258 (-11%) and K (-9%) concentrations as opposed to maize fertilized with NPK mineral
259 fertilizer, probably resulting from the lower K_2O amount provided by the pellets v. the NPK
260 fertilizer (60, 110 and 200 by SSFC, WCC and NPK fertilizer, respectively). All treatments
261 produced similar TN levels.

262 Maize N and P concentrations were significantly ($P = 0.009$ and $P \leq 0.001$ for N and P,
263 respectively) influenced by characteristics of the pellet applied, as demonstrated by
264 increased N concentrations in WCC relative to SSFC. In the case of P, plants fertilized with
265 SSFC had the highest concentrations. A significant ($P < 0.001$) rise in N concentration was
266 induced in WCC with smaller- as opposed to larger-sized pellets (6 v. 8 mm), although no
267 such effect was detected in SSFC. Application method significantly ($P \leq 0.001$, < 0.001 and
268 ≤ 0.001 for SSFC 8, WCC 6 and WCC 8, respectively) influenced N concentration, as
269 evidenced by increased N concentrations when pellets were mixed into the soil as opposed
270 to surface-applied.

271 With regards to P and K, the higher P content in SSFC played a key role in increasing maize
272 P concentration, while the high K content in WCC did not produce such an effect on the
273 plant. Neither pellet application method nor dimension produced any important P or K effect.
274 Alternatively, if an effect was indeed produced, it might have been countered by different
275 interactions.

276 Apparent recovery of both N and P were affected by treatment (Table 5). No significant effect
277 of pellet type relative to NPK fertilizer or of SSFC relative to WCC was detected. However,
278 soil incorporation improved the AR of N in every tested situation and the same was observed
279 for the small diameter relative to the large one. The AR of P was lowered by the use of
280 pellets compared with mineral fertilizer and by WCC compared with SSFC. Soil incorporation
281 affected the AR of P, but in dissimilar ways for pellet type and diameter. Small-sized pellets
282 improved the AR of P only for SSFC, but not for WCC.

283 Root production ($P < 0.001$) and N concentration ($P < 0.001$) were affected significantly by
284 treatments (Table 3), with all fertilized treatments producing a significantly greater root
285 biomass ($P < 0.001$) (Table 6) compared with the control. No significant differences were
286 observed between mineral fertilizer and pellets. Root production was stimulated when no
287 bulking agent was used in the composting process, an effect that was significantly greater
288 when SSFC was mixed into the soil. Smaller diameter pellets also positively affected root
289 production.

290 After 65 days, highly significant differences in root N concentration were observed
291 comparing the unfertilized control with respect of all the other treatments ($P \leq 0.001$). After
292 the same period, root N concentrations were lower for pellet-fertilized treatments than for
293 NPK treatment ($P = 0.004$).

294

295 **Soil properties**

296 No significant differences were found in soil pH, while significant treatment effects were
297 detected for NO_3^- ($P < 0.001$) and EC ($P < 0.001$) (Table 3). In particular, NO_3^- and EC
298 increased after the application of NPK mineral fertilizer with respect to pellets (Table 7). Both
299 soil properties were affected by the type of pellet supplied and, specifically, they increased
300 in WCC treatment. In addition, pellet diameter had an important effect on NO_3^- and EC: in
301 general, the highest values were observed with 6 mm pellets. Statistical analysis highlighted

302 that the superficial distribution promotes the increase of NO_3^- and EC. In all treatments
303 investigated (Table 7), EC values were well below the limit for saline soil and, furthermore,
304 soil in all the treatments can be considered non-saline (Bernal et al. 1992).
305 Soluble-N analysed in soil pore water indicated an important effect of sampling time, with
306 the lowest concentration at the end of the experiment in all treatments (Fig. 2). With respect
307 to the treatments, results of NPK fertilizer at the first sampling was statistically greater
308 ($P < 0.001$) than the rest of the treatments, including unfertilized control.

309

310 **Ammonia volatilization and GHG emissions**

311 Ammonia volatilization was not detected from any of the treatments investigated (data not
312 shown). Methane emission measurements were low and unaffected by the various
313 treatments (data not shown), while the various treatments showed significant influence on
314 CO_2 ($P < 0.001$) and N_2O ($P = 0.002$) emissions (Table 3).

315 During the 57-day incubation period, the unfertilized control showed the lowest CO_2
316 emission (Table 8). No significant differences were found between SSFC and WCC
317 treatments. However, across the SSFC treatments, significant differences ($P = 0.007$) were
318 observed between the two pellets diameter sizes, with higher CO_2 emissions recorded for
319 the smaller diameter pellet. Other differences were not significant.

320 All fertilized treatments exhibited cumulative N_2O emissions significantly higher than the
321 control ($P = 0.002$) (Table 8). Cumulative N_2O emissions were not significantly affected by
322 pellet type or pellet diameter; however, the statistical analysis revealed that superficial
323 distribution reduced N_2O emissions.

324 **DISCUSSION**

325 Pig slurry SF has been investigated in its pelletized form after composting as a fertilizer for
326 maize crop, a technique proposed to add agronomic value while mitigating the
327 environmental risk of conventional SF pig slurry. A set of follow-on trials tested compost

328 type, pellet size and application method to identify optimizations of the technique. To this
329 end, four hypotheses were developed. The first hypothesis tested whether compost derived
330 from SF pig manure possessed a short-term significant fertilizer effect beyond that of its
331 value as an amendment. The current investigation verified that the SF pig slurry pelletized
332 compost fertilizers considered effectively increased maize biomass, NPK concentration and
333 root N content, as well as residual soil nitrates and EC in all treatments fertilized with
334 compost pellets, compared with the unfertilized control. The results obtained are consistent
335 with the acknowledgement that composted SF pig slurry is an improved fertilizer product,
336 mainly due to its large contribution of nutrients to plants, especially N and P (Pinamonti et
337 al. 1997; Atiyeh et al. 2001; Garcia-Gomez et al. 2002; Perez-Murcia et al. 2006). However,
338 lower N concentrations of aerial and root biomasses, K concentrations in maize plants and
339 residual soil nitrates in all pellet-fertilized treatments v. the NPK mineral fertilizer treatment
340 were observed. The results obtained highlighted that pelletized treatments provided lower –
341 possibly even inadequate – amounts of K during the growing season relative to mineral
342 fertilizer, a finding consistent with the lower yields produced in maize fertilized with compost,
343 compared with mineral fertilizer (Businelli et al. 1990; Bazzoffi et al. 1998; Loecke et al.
344 2004).

345 The results of the current investigation also confirmed that pig manure compost pellet
346 fertilizers released N slowly when compared with standard NPK-soluble mineral fertilizer. In
347 fact, the analysis of soil pore water during the experiment indicated a similar behaviour of
348 soluble N (readily plant-available) in all pellet treatments, but the greater concentration found
349 in the first sampling of NPK-fertilizer demonstrated the high solubility of the mineral fertilizer
350 with respect to pelletized compost. At the end of the experiment, the results showed that
351 soluble N was taken up by the crop in all treatments. As Ball et al. (2004) pointed out, the
352 slower nutrient release of pelletized compost over time can act to reduce the risk of nutrient
353 losses significantly. Efficiency of added N, as estimated through apparent recovery (NAR),

354 was not statistically lower for pellets than for NPK. This difference was not determined by
355 different yield rather than by different N concentration in plant, determining an improvement
356 of uptake in NPK treatment. Small diameter (6mm) pellets v. large (8 mm) pellets were
357 shown to improve NAR values, which advances the notion that pellets did not threaten maize
358 yield performances relative to mineral fertilizer, only that they may reduce the nutritional
359 value of maize destined for feed purposes. It is also possible that the added N not used by
360 plants and not present in the soil in mineral form at the end of the cropping cycle remained
361 in the soil in stable pools as organic-N to improve soil fertility over time (Zavattaro et al.
362 2016), or was lost through leaching or gaseous emissions. Nonetheless, the fact that mixing
363 pellets (6 mm diameter) into the soil resulted in improved NAR values relative to surface
364 application make the second hypothesis feasible for NH₃ volatilization.

365 For P, the AR of applied fertilizer is usually low in the first cropping year following application,
366 when as much 0·90 of added inorganic P has been shown to become unavailable for crop
367 nutrition due to adsorption and precipitation (Malik et al. 2012). The results obtained in the
368 current study followed this trend also, with a range of 'very low' AR values (from 0·10 to
369 0·18). Even though statistical effects were identified, differences failed to permit conclusions
370 on the fertilizer value of using pelletized com- posts for P nutrition.

371 The second hypothesis tested whether adding a bulking agent before composting failed to
372 limit the fertilizer properties of SF-based pelletized composts. It too was verified. The results
373 of the planned contrast test between SSFC and WCC highlighted that indeed no differences
374 were found in plant yields, plant K concentrations, root N concentrations, or NAR values.
375 Moreover, plant N concentration increased when WCC was applied, which suggested an
376 improvement in availability of mineral N to plants. Increased maize root biomass was
377 measured in SSFC relative to WCC, a result that might have been induced by lower nutrient
378 availability and a subsequent increase in root allocation (Müller et al. 2000). The supposition
379 of high availability of nutrients in WCC is further corroborated by increased residual soil

380 nitrates found after application of WCC instead of SSFC. These results demonstrate that the
381 addition of a bulking agent during composting fails to reduce the fertilizer value.

382 The third hypothesis of the current study tested whether reduced pellet diameter resulted in
383 increased NPK availability, as well as concurrently NH_3 volatilization and GHG emissions.
384 The hypothesis was partially verified. In the WCC treatment, the results of the planned
385 contrast test of 6 v. 8 mm indicated that smaller diameter pellets induced increased plant N
386 concentration and root production, in addition to soil EC, residual soil nitrates and NAR. The
387 larger diameter increased plant P concentration alone. In SSFC treatment, the smaller
388 diameter resulted in increased root production, soil EC and residual soil nitrates. Carbon
389 dioxide emissions also increased, which others (Rochette et al. 2000; Balota et al. 2010)
390 ascribe to the raised soil microbial activity when pellet diameter is smaller and the applied
391 OM more degradable.

392 The last hypothesis postulated that incorporating compost pellets into the soil reduces NH_3
393 volatilization and GHG emissions and simultaneously increases nutrient availability. This
394 hypothesis was partially verified. The planned contrast test of mixed v. surface application
395 highlighted that incorporating pellets into the soil greatly affected plant N concentration, root
396 production, NAR and soil residual nitrates (reduction). Following application, each of these
397 measures demonstrated that plant N uptake was improved except in the case of root
398 production. Considering GHG emissions, soil mixing did not affect CO_2 emissions, but
399 induced an increase in N_2O emissions as expected from the higher contact of fertilizer with
400 soil particles and enhanced microbial degradation (Velthof et al. 2003). Surface application
401 played a different role in nutrient release dynamics by reducing nutrient availability to the
402 plant, while simultaneously, increasing residual nitrates in the soil. This behaviour may be
403 explained by late transfer of added N from the surface toward the soil (retarded or reduced
404 solubilization of pellets) that was unmatched by plant requirements. Soil incorporation is the
405 best technique to take advantage of the nutrients available from pellets.

406 **CONCLUSIONS**

407 Pelletized composted manure was shown to be an effective slow-release fertilizer for maize.
408 The best technical options for its production include addition of a bulking agent before
409 composting, using small diameter pellets and application with incorporation into the soil. The
410 adoption of all these techniques results in the best availability of nutrients from pelletized
411 composted pig manure for plant nutrition.

412

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545

546 **Table captions**

547 **Table 1.** Main properties of the two types of pellet included in the experiment.

548 **Table 2.** Basic chemical properties of the soil used in the experiment.

549 **Table 3.** Results of ANOVA of all measured variables. Significance of the treatment and
550 Standard Error of the Mean (SEM) of the treatments.

551 **Table 4.** Effects of the fertilization treatments on maize production and nutrient content.

552 **Table 5.** Effects of the fertilization treatments on root production and its N content.

553 **Table 6.** Effects of the fertilization treatments on Apparent Recovery of N and P.

554 **Table 7.** Effects of the fertilization treatments on residual soil quality.

555 **Table 8.** Effects of fertilization treatments on CO₂ and N₂O emissions.

556

557 **Table 1. Main properties of the two types of pellet included in the experiment.**

Parameter	SSFC (Ø 6 mm and Ø 8 mm)		WCC (Ø 6 mm and Ø 8 mm)	
	Average	S.E.	Average	S.E.
Dry Matter (%)	85.4	0.7	84.6	0.4
Moisture (%)	14.6	0.7	15.4	0.4
pH	8.1	0.1	7.9	0.1
TN (%)	3.3	0.1	2.9	0.1
NH ₄ ⁺ -N (mg kg ⁻¹)	672.0	10.5	495.8	17.7
NO ₃ ⁻ -N (mg kg ⁻¹)	1460.0	13.8	2390.0	13.8
TOC (%)	36.9	0.4	38.1	0.2
C/N	11.2	0.3	13.2	0.3
OM (%)	63.6	1.5	65.7	0.5
CEC (cmol kg ⁻¹)	70.9	1.7	79.5	4.2
P ₂ O ₅ (%)	4.0	0.1	3.7	0.2
K ₂ O (%)	1.0	0.1	1.6	0.1

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560 **Table 2. Chemical properties of the soil used in the experiment.**

PARAMETER	AVERAGE	S.E.
pH	8.55	0.01
EC (dS m ⁻¹)	0.18	0.01
WHC (%)	31.50	1.02
CaCO ₃ (%)	38.70	0.40
CEC (cmol kg ⁻¹)	10.50	0.50
OM (%)	0.88	0.03
TOC (%)	0.51	0.01
TN (%)	<0.01	<0.01
C/N	7.29	0.09
NH ₄ ⁺ -N (mg kg ⁻¹)	10.8	0.80
Available-P (mg kg ⁻¹)	27.7	0.50

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563 **Table 3. Results of analysis of variance (ANOVA) of all measured variables**

Parameters	Treatment P (f)	SEM
plant yield (g D.M. pot ⁻¹)	0.010	0.865
plant N (% D.M.)	0.000	0.065
plant P (g kg ⁻¹ D.M.)	0.000	0.091
plant K (g kg ⁻¹ D.M.)	0.000	0.819
root production (g D.M. pot ⁻¹)	0.000	0.123
root N (%D.M.)	0.000	0.068
soil NO ₃ (mg kg ⁻¹ soil)	0.000	0.3844
soil EC (μS cm ⁻¹)	0.000	3.936
soil pH	0.310	0.038
cumulative CO ₂ (mg C-CO ₂ m ⁻²)	0.000	4.211
cumulative N ₂ O (mg N-N ₂ O m ⁻²)	0.002	110.370
cumulative CH ₄ (mg C-CH ₄ m ⁻²)	0.652	0.983

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565 **Table 4. Effects of fertilization treatments on maize production and nutrient content**

In	Contrast			Plant yield (g D.M. pot ⁻¹)			Plant N (% D.M.)			Plant P (g kg ⁻¹ D.M.)			Plant K (g kg ⁻¹ D.M.)		
	1	vs	2	Average 1	Average e 2	P(F)	Average e 1	Average 2	P(F)	Average 1	Average e 2	P(F)	Average e 1	Average 2	P(F)
ALL	CONTROL	vs	FERTILISED	12.57	16.31	0.000	1.28	1.91	0.000	1.60	1.98	0.000	27.92	34.74	0.000
FERTILISED	NPK	vs	PELLET	15.62	16.39	0.411	2.12	1.89	0.002	1.96	1.99	0.771	37.70	34.36	0.001
PELLET	SSFC	vs	WCC	16.93	15.85	0.088	1.82	1.95	0.009	2.10	1.87	0.001	34.17	34.56	0.498
SSFC	6	vs	8	17.52	16.34	0.181	1.83	1.81	0.731	2.19	2.02	0.069	34.08	34.25	0.834
SSFC 6	Surface	vs	Mixed	17.32	17.72	0.746	1.75	1.91	0.092	2.47	1.91	0.000	33.26	34.90	0.169
SSFC 8	Surface	vs	Mixed	15.25	17.42	0.087	1.64	1.99	0.001	1.96	2.07	0.390	36.11	32.40	0.003
WCC	6	vs	8	16.50	15.20	0.144	2.16	1.75	0.000	1.74	2.00	0.007	34.67	34.46	0.800
WCC 6	Surface	vs	Mixed	16.25	16.75	0.686	1.97	2.35	0.000	1.51	1.96	0.002	32.72	36.62	0.002
WCC 8	Surface	vs	Mixed	14.92	15.47	0.657	1.57	1.92	0.001	2.19	1.82	0.009	33.84	35.08	0.292

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568 **Table 5. Effects of fertilization treatment on apparent recovery of nitrogen (N) and phosphorus (P)**

In	Contrast		N Apparent Recover (% of added N)			P Apparent Recover (% of added P)		
	1	2	Average 1	Average 2	P(F)	Average 1	Average 2	P(F)
FERTILISED	NPK	vs PELLET	86,0	75,3	0,051	15,4	13,3	0,002
PELLET	SSFC	vs WCC	74,8	75,8	0,767	14,8	11,8	0,000
SSFC	6	vs 8	81,2	68,4	0,016	16,0	13,6	0,000
SSFC 6	Surface	vs Mixed	44,4	92,5	0,000	12,4	14,8	0,008
SSFC 8	Surface	vs Mixed	72,6	89,8	0,021	17,8	14,2	0,000
WCC	6	vs 8	98,6	53,1	0,000	11,4	12,2	0,165
WCC 6	Surface	vs Mixed	37,3	68,9	0,000	13,1	11,3	0,036
WCC 8	Surface	vs Mixed	80,0	117,1	0,000	9,8	13,0	0,001

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571 **Table 6. Effects of fertilization treatment on root production and its nitrogen content.**

In	Contrast			Roots production (g D.M. pot ⁻¹)			Roots N (% D.M.)		
	1	2		Average 1	Average 2	P(F)	Average 1	Average 2	P(F)
ALL	CONTROL	vs	FERTILISED	1.37	2.04	0.000	0.77	1.04	0.001
FERTILISED	NPK	vs	PELLET	1.97	2.12	0.110	1.23	1.01	0.004
PELLET	SSFC	vs	WCC	2.33	1.90	0.000	1.04	0.99	0.304
SSFC	6	vs	8	2.55	2.11	0.001	1.11	0.97	0.060
SSFC 6	Surface	vs	Mixed	2.12	2.97	0.004	1.02	1.19	0.103
SSFC 8	Surface	vs	Mixed	1.85	2.37	0.000	0.87	1.07	0.053
WCC	6	vs	8	2.15	1.66	0.000	0.99	0.99	1.000
WCC 6	Surface	vs	Mixed	2.12	2.17	0.776	0.95	1.03	0.396
WCC 8	Surface	vs	Mixed	1.55	1.77	0.140	0.91	1.06	0.137

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Table 7. Effects of the fertilization treatments on residual soil quality

In	Contrast			Soil NO ₃ (mg kg ⁻¹ soil)			Soil EC (dS m ⁻¹)		
	1		2	Average 1	Average 2	P(F)	Average 1	Average 2	P(F)
ALL	CONTROL	vs	FERTILISED	6.72	11.15	0.000	0.304	0.318	0.002
FERTILISED	NPK	vs	PELLET	12.34	10.00	0.003	0.334	0.316	0.000
PELLET	SSFC	vs	WCC	10.60	11.40	0.004	0.311	0.321	0.002
SSFC	6	vs	8	11.81	10.39	0.030	0.335	0.308	0.000
SSFC 6	Surface	vs	Mixed	13.82	9.81	0.000	0.346	0.323	0.000
SSFC 8	Surface	vs	Mixed	12.42	8.35	0.000	0.312	0.303	0.095
WCC	6	vs	8	12.74	10.05	0.000	0.326	0.297	0.000
WCC 6	Surface	vs	Mixed	15.10	10.37	0.000	0.345	0.307	0.000
WCC 8	Surface	vs	Mixed	10.45	9.66	0.155	0.292	0.302	0.087

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577 **Table 8. Effects of fertilization treatments on carbon dioxide (CO₂) and nitrous oxide (N₂O) emissions.**

In	Contrast		Cumulative CO ₂ (mg C m ⁻²)			Cumulative N ₂ O (mg N m ⁻²)		
	1	2	Average 1	Average 2	P(F)	Average 1	Average 2	P(F)
ALL	CONTROL	vs PELLET	1212.9	1676.9	0.001	-1.48	14.08	0.002
PELLET	SSFC	vs WCC	1740.2	1613.5	0.118	14.92	13.24	0.576
SSFC	6	vs 8	1902.6	1577.8	0.007	16.63	13.21	0.425
SSFC 6	Surface	vs Mixed	1767.8	2037.4	0.097	4.13	29.13	0.000
SSFC 8	Surface	vs Mixed	1554.0	1601.6	0.730	5.06	21.37	0.011
WCC	6	vs 8	1704.4	1522.8	0.113	12.60	13.87	0.765
WCC 6	Surface	vs Mixed	1673.4	1735.3	0.695	0.63	24.57	0.000
WCC 8	Surface	vs Mixed	1394.2	1651.3	0.113	5.55	22.19	0.010

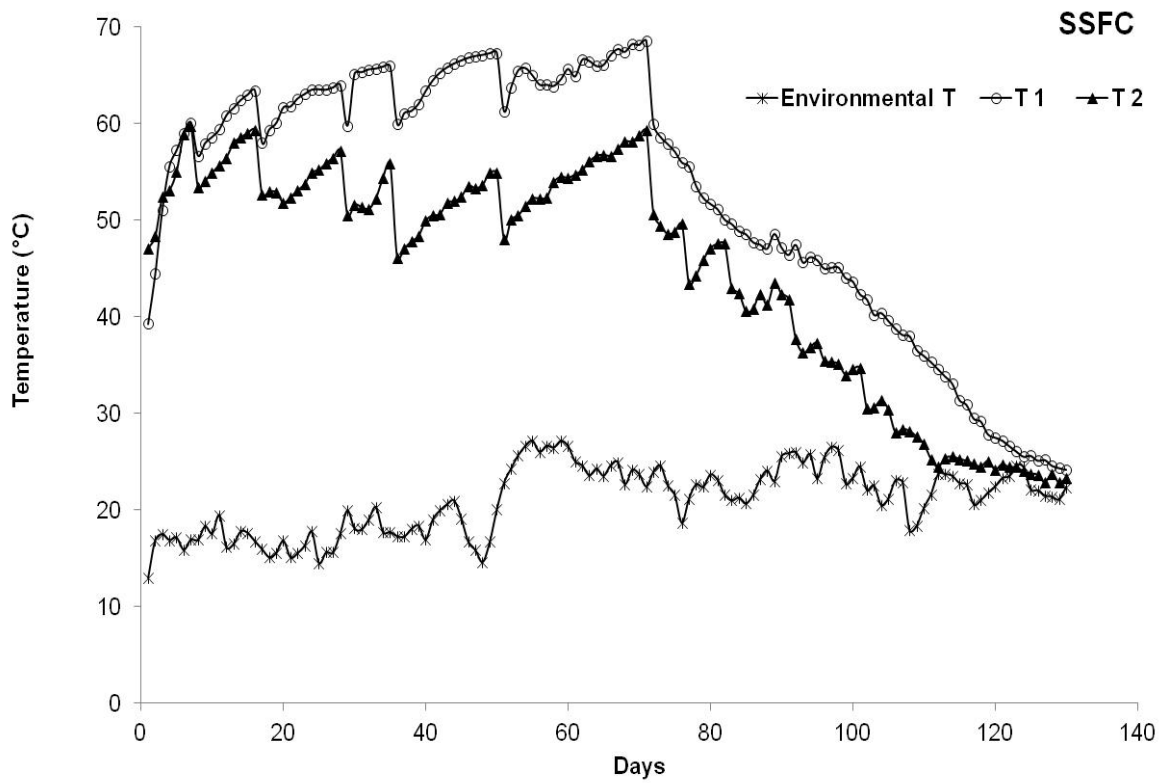
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579 **Figure captions**

580 **Figure 1.** Temperature trends (°C) recorded during the composting trial (daily average).

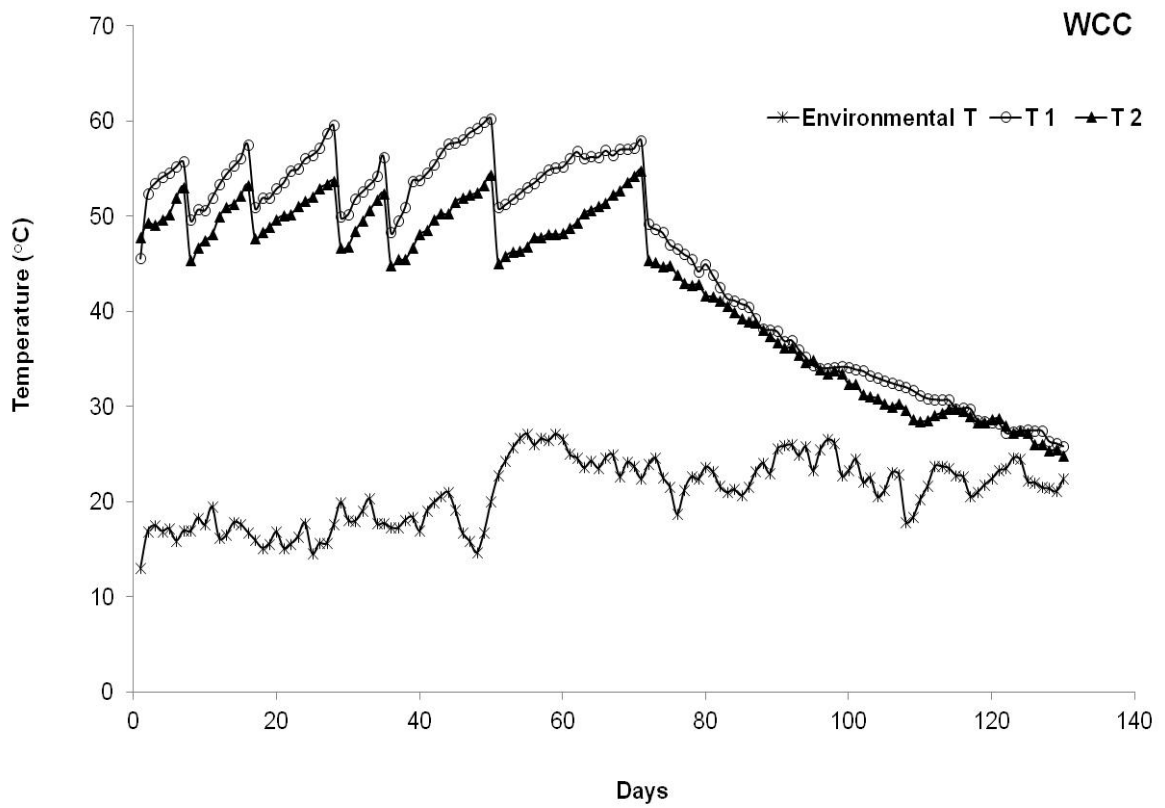
581 **Figure 2.** Concentration of soluble nitrogen in pore water soil at different treatment with
582 pellets mixed with the soil, nitrogen: phosphorus: potassium (NPK) fertilizer and unfertilized
583 control during the mesocosm experiment.

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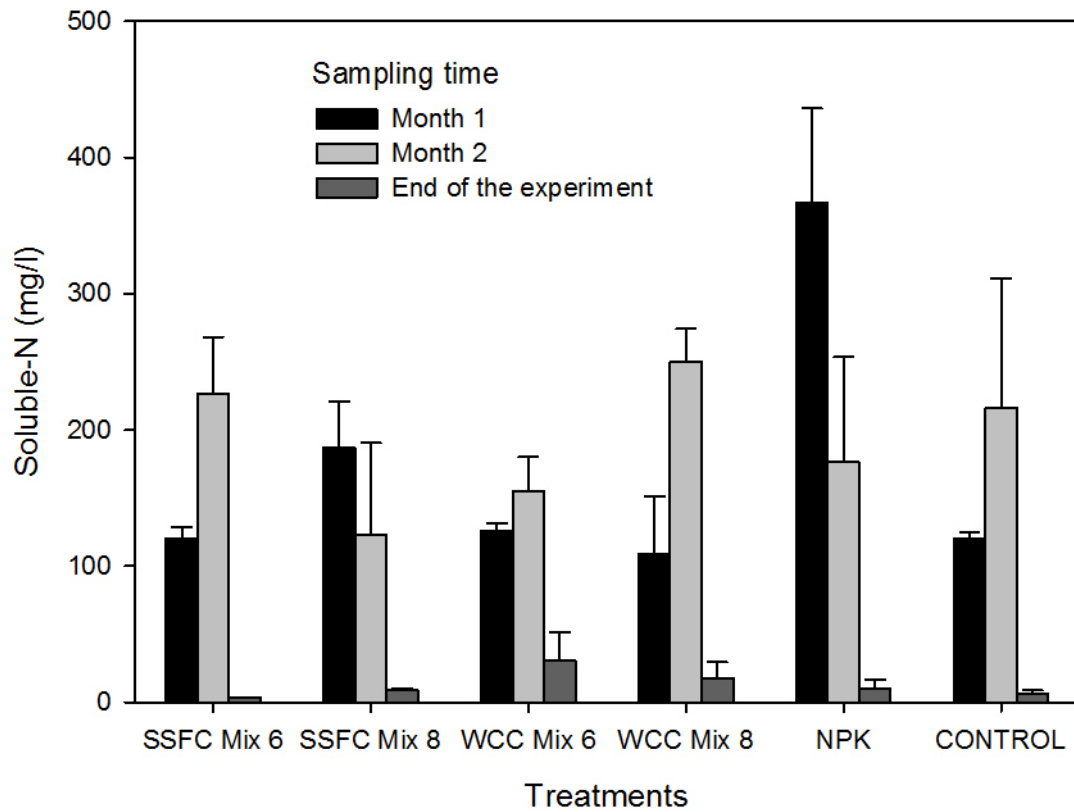


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588 **Fig. 1. Temperature trends (°C) recorded during the composting trial (daily average).**

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592 **Fig. 2. Concentration of soluble nitrogen in pore water soil at different treatment with pellets mixed**
593 **with the soil, nitrogen: phosphorus: potassium (NPK) fertilizer and unfertilized control during the**
594 **mesocosm experiment.**

595