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12 *Running title: Effects of slurry management on soil aggregates*

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16 **Summary**

17 Effects of applications of pig slurry on soil aggregate stability are not well
18 understood in dryland agriculture. This research aims to (i) identify aggregate
19 stability tests that give a reliable description of the soil's behaviour when pig
20 slurry (PS) is applied to calcareous soil and (ii) interpret them in terms of
21 chemical, biological, morphological and physical soil properties for soil quality
22 assessments. Soil samples from eight fertilizer treatments (mineral fertilizers and
23 PS), applied over seven growing seasons were analysed. We applied five
24 methodologies to examine different mechanisms of aggregate breakdown.
25 Porosity was characterized by image analyses. There was minimum resistance to
26 the mechanical breakdown of aggregates when slurries were applied 12 months
27 before analysis. Recent applications of slurry (3 months before the analysis)
28 improved resistance to implosion caused by the penetration of water into dry
29 aggregates (slaking), although the opposite result can occur if the method of
30 evaluation is not chosen properly. Recent applications of PS also enhanced soil
31 respiration and increased soil porosity in the 25–100 μm size range (packing
32 pores between aggregates) and in the 100–400 μm size range (interaggregate or
33 faunal pores). In dryland systems and in the winter cereal cropping season, the
34 resistance of dry aggregates to slaking is improved temporarily if PS is applied
35 at N rates equivalent to around 1.7 Mg OM ha⁻¹ year⁻¹.

36 *Keywords: Aggregate breakdown dynamics, arid soil, pore-size distribution, soil*
37 *fertility, soil respiration.*

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39 **Highlights**

- 40 • How does soil aggregate strength change in response to pig slurry (PS) applications?
- 41 • Improve understanding of PS as a fertilizer and its effect on soil quality assessments
- 42 • Changes in aggregate stability are controlled by time since pig slurry application
- 43 • The effects of PS on the increment of packing pores between aggregates are transient
- 44
- 45

46 **Introduction**

47 The stability of soil aggregates and architecture of the pore space affect the movement
48 and storage of water, aeration, erosion, biological activity and the growth of crops. The
49 chief mechanisms of aggregate breakdown by water (Le Bissonnais, 1996) are: (i)
50 compression of air entrapped inside aggregates during wetting, which occurs when dry
51 aggregates are immersed in water or rapidly wetted, (ii) breakdown by microcracking of
52 aggregates because of internal pressure from differential swelling of clays, (iii)
53 mechanical breakdown of aggregates by external pressures caused by raindrop or other
54 impacts and (iv) physicochemical dispersion caused by changes in the double-layer
55 forces that act between electrically charged colloids. Slaking and differential swelling
56 induce changes in soil structure because they result in the breakdown of larger
57 macroaggregates to smaller microaggregates. Raindrop impact and physicochemical
58 dispersion may cause the release of soil particles. Every cause of aggregate breakdown
59 requires different methodologies to measure it. Some differences arise from the swelling
60 potential of different soil minerals (Emerson, 1964) because the clay fraction in natural
61 aggregates usually consists of a mixture of minerals of various particle sizes. In
62 semiarid environments, the presence of carbonates interferes with the relation between
63 clay minerals and soil aggregation, resulting in enhanced macroaggregate stabilization
64 in carbonate-rich soil but with less porosity within the macroaggregates (Virto *et al.*,
65 2013). Furthermore, because calcium is adsorbed better to clay surfaces than is sodium,
66 it reduces swelling and dispersion, which preserves structural integrity. The type of clay
67 minerals also affects the proportion of air and water in an aggregate. The air-filled
68 porosity of aggregates dominated by kaolinite and illite is larger than that of aggregates
69 dominated by smectite (Oades, 1986), which also implies greater hydraulic conductivity

70 in the former. Kaolinite and illite have more rigid structures than smectite, and the stress
71 induced by the rapid entrapment of air inside aggregates containing kaolinite and illite
72 facilitates slaking (Tessier *et al.*, 1990). Slaking is also affected by the initial water
73 content. Slaking decreases as the initial water content of aggregates increases until
74 saturation is reached (Panabokke & Quirk, 1957).

75 Size distribution, quantity and stability of aggregates determine the amount and
76 distribution of pore spaces associated with them. Soil pore characteristics are required
77 for the complete assessment of habitat availability for micro-fauna and microorganisms,
78 of the aptitude for root growth, of fluid transport through the soil profile and of soil
79 aeration. Lynch & Bragg (1985) suggested that pores of 0.1–15 μm hold water available
80 to plants, pores larger than 30–60 μm allow water to drain under gravity and those >60
81 μm are important in gas exchange. The pore spaces affect almost everything that occurs
82 in the soil; they are affected by particle-size distribution, soil structure and forces that
83 disrupt this. Micromorphology techniques with 2-D images from undisturbed soil thin
84 sections enable us to study the porosity of the 3-D soil body because some stereological
85 relations exist, such as the one that equates pore volume density to their areal extent in
86 2-D (Ringrose-Voase & Nortcliff, 1987).

87 There is clear evidence that microorganisms are involved in the aggregation process
88 because microbial activity controls the production of exudates which act as binding
89 agents in aggregates (De Gryze *et al.*, 2005). In any soil where clay is present,
90 interactions between the polysaccharide exudates, organic colloids and other products
91 of decomposition promote stability (Dontsova & Bigham, 2005). Soil structure is
92 improved by the addition of organic materials to soil (Tisdall & Oades, 1982).
93 Nevertheless, detailed information about the effects of pig (*Sus scrofa domesticus*)
94 slurries on soil physical properties is lacking. This is an important issue in Spain

95 because it is the leading European country for pig production, and pig slurries are often
96 applied directly to fields each year, mainly in dryland farming systems. Furthermore,
97 slurries of different origin have different composition; the main differences are between
98 slurries from fattening pigs and sows (Yagüe *et al.*, 2012a).

99 Apart from its OM content, pig slurry (PS) can affect soil structure in various ways.
100 Slurries supply Na, Mg and Ca in addition to the three major nutrients (N, P and K).
101 The exchangeable cations (K^+ , Na^+ , Mg^{2+} and Ca^{2+}) affect aggregation. Sodium (Na^+)
102 causes mainly swelling or dispersion or both of clay particles and slaking of unstable
103 aggregates (Crescimanno *et al.*, 1995). Aggregate dispersion generally leads to rapid
104 crusting of the soil, slow infiltration and great mobility of particles in water. In
105 calcareous soil (with exchangeable sites saturated with calcium), both Ca^{+2} and Mg^{+2}
106 reduce clay dispersion (Amézketa & Aragües, 1995). Furthermore, the adsorbed K^+
107 limits clay dispersion because of its hydration energy, which is equivalent to 72% of
108 that of adsorbed Na^+ (Levy & Torrento, 1995).

109 The main goal of this study was to assess methodologies that can reflect potential
110 changes in aggregate stability with the application of PS to calcareous soil. We began
111 with the hypothesis that success in quantifying potential changes in aggregate stability
112 after a medium period (seven growing seasons) of PS use: (i) will be closely linked to
113 the dominant breakdown mechanism taking place, (ii) the best evaluation method
114 should be supported by other soil properties such as chemical (cation exchange
115 capacity, CEC), biological (soil respiration) and physical (pore-size distribution from
116 image analyses in spite of the scale differences between aggregates or the soil fraction,
117 and porosity in the bulk soil) and (iii) the differences between the methods could be
118 affected by the length of sampling time since the last slurry application.

119 The choice of methods took into account the different mechanisms of aggregate
120 breakdown and the factors affecting them, such as the method of wetting. Although the
121 aggregate stability tests were designed mainly to compare different types of soil or
122 climatic conditions for a given soil (Le Bissonnais, 1996), we applied them to compare
123 time-dependent changes associated with fertilizer management.

124 **Materials and methods**

125 *Site description*

126 The experiment was in the Ebro valley (NE Spain). The area has a Mediterranean
127 semiarid climate with an average annual precipitation of 436 mm for the period 2000–
128 2010. The altitude is 443 m a.s.l. and the coordinates are 41° 52' 29" N, 1° 09' 10" E.
129 The soil is classified as a Typic Xerofluvent (Soil Survey Staff, 2014) and the surface
130 horizon (0–0.30 m) had a pH of 8.2 (1:2.5 soil:distilled water), organic matter content of
131 20.1 g kg⁻¹, CEC was 11.1 cmol_c kg⁻¹ and the texture is a silty loam (131 g kg⁻¹ sand, 609
132 g kg⁻¹ silt and 260 g kg⁻¹ clay). Illite and chlorite are the dominant clay minerals with
133 traces of talc and 1:1 clays such as kaolinite (Figure 1). The soil is not saline and
134 calcium carbonate (CaCO₃) content was 300 g kg⁻¹.

135 The experimental site was established in 2002 and winter cereals were grown until
136 2011, except for the 2007–08 cereal growing season when the field was left fallow. The
137 rotation was: three years barley (*Hordeum vulgare* L.) and one year wheat (*Triticum*
138 *aestivum* L.). Sowing was done between late October and early November, and after
139 harvest (end of June to early July) straw was removed from the fields for animal
140 bedding and feed.

141 The experimental area was divided into three blocks and seven fertilizer treatments
142 were assigned at random to the plots in each block. These treatments yielded an average

143 biomass (at 0% humidity) that ranged from 2985 to 3543 kg grain ha⁻¹ year⁻¹. One
144 control (no N, no OM applied) with an average grain yield (0% humidity) of 2624 kg
145 ha⁻¹ year⁻¹ was also included to give eight treatments in total (Table 1). Apart from this
146 control, one treatment received only mineral fertilizer and the rest (six treatments)
147 included PS of different origins, either fattening pigs (FS) or sows (SS). Just before
148 sowing, four treatments received 30 t ha⁻¹ year⁻¹ of slurry from fattening pigs (30FS),
149 the remaining four did not receive slurry or mineral nitrogen (0). In February (at the
150 cereal tiller growth stage), the soil of six of the treatments received fertilizer. Two of
151 them received mineral nitrogen fertilizer (M): 60 or 120 kg N ha⁻¹ year⁻¹ (60M or
152 120M), two received slurry from fattening pigs: 20 or 60 t ha⁻¹ year⁻¹ (20FS or 60FS)
153 and the last two received slurry from sows: 60 or 90 t ha⁻¹ year⁻¹ (60SS or 90SS). In the
154 two treatments where slurry was applied only at tillering (0–60FS or 0–90SS, rates at
155 sowing and tillering, respectively), the average range of OM applied (1764–2622 kg ha⁻¹
156 year⁻¹) was similar to the range applied (1681–2491 kg ha⁻¹ year⁻¹) to plots that
157 received slurry only at sowing (30FS–0 or 30FS–60M, rates at sowing and tillering,
158 respectively) or combined with slurry at tillering (30FS–20FS or 30FS–60SS, rates at
159 sowing and tillering, respectively).

160 Apart from yield records, the treatments were chosen because they enabled
161 comparisons between the amounts of OM applied and between the different application
162 times. Treatments 0–90SS and 30FS–0 had similar average amounts of OM applied
163 over the seven growing seasons (Table 1). These rates of OM applications were also
164 increased by 50% in two other treatments (0–60FS and 30FS–20FS). Finally, we added
165 an intermediate rate of treatment of 30FS–60SS with an increment of 25% in the OM
166 applied.

167 Slurries were applied by the splash plate method. The control and the plots with
168 only mineral N fertilizer (0–120M, rate at sowing and rate at tillering, respectively)
169 received mineral phosphorus (42 kg P ha⁻¹ year⁻¹) and potassium (89 kg K ha⁻¹ year⁻¹)
170 before sowing.

171 All fertilizers applied just before sowing were incorporated into the soil and mixed
172 with the stubble remaining in the field by tillage with an offset disc harrow (0–0.15 m).
173 This incorporation favours a uniform distribution of slurries at depth. The size of plots
174 that had pig slurry was 274 m² (11-m wide and 25-m long) and the size of the control
175 plots and plots with mineral fertilizer only (0–120M) was 174 m² (7-m wide and 25-m
176 long).

177 *Soil sampling, chemical and biological soil analysis*

178 Soil sampling was done two days before top-dressing on 23 February 2011 (after 7
179 years of fertilizer treatments and one of fallow). This means that four treatments were
180 sampled three months after the last slurry application (S3mo) and two treatments were
181 sampled twelve months after the last slurry application (S12mo). From each treatment
182 and block, soil samples were taken at 0–10-cm depth with a corer (7 cm in diameter
183 with steel bores) to assess aggregate stability and biological activity, and for chemical
184 analysis. For each plot (associated with a specific treatment), four points were sampled.
185 Part of each sample was air-dried and stored, and the rest was refrigerated before
186 processing. All samples were sieved gently to 2 mm before being analysed.

187 Furthermore, undisturbed soil samples from a similar depth to the above were taken
188 to study porosity from soil thin sections. From each treatment, three undisturbed soil
189 samples with a rectangular prism shape (6.2 cm × 9.1 cm × 19.3 cm) were taken from
190 the three replicate blocks to give 72 samples or rectangular prisms. The undisturbed

191 rectangular prisms were dried at room temperature and impregnated with polyester resin
192 with a fluorescent dye (Uvitex©, CibaGeigy Ltd, Manchester, England). One vertical
193 thin section (50-mm wide, 130-mm long) was made from each prism. Pore morphology
194 was evaluated qualitatively.

195 From each thin section, three fields (31.5-mm wide, 42.0-mm long) were selected to
196 obtain images with an Olympus® C-7070 wide zoom camera (Olympus Corporation,
197 Tokyo, Japan) under three light conditions: parallel polarizers (PPL), crossed polars
198 (XPL) and incident UV light. The latter was processed with ImageJ (Rasband, 2008) to
199 obtain digital binary images (with PPL and XPL as control) from which the total
200 porosity was analysed statistically. Each image set was used to analyse pore-size
201 distribution based on an ‘opening’ algorithm of mathematical morphology with the
202 Quantim4 library (Vogel, 2008). Pore sizes in the following four ranges (values in
203 micrometres): 25–65, 65–100, 100–200, 200–400 and > 400 μm were identified.

204 Cation exchange capacity and exchangeable cations were evaluated (for each
205 treatment and block) by extraction with ammonium acetate 1N (pH=7) in a composite
206 sample (obtained from the four sampled points) of each plot. The exchangeable cations
207 K^+ , Na^+ , Mg^{2+} and Ca^{2+} were determined following Hendershot *et al.* (2008).

208 Microbial activity and aggregate stability procedures required three and four
209 replicates, respectively, to be analysed for each plot and it should be evaluated as soon
210 as possible after sampling. Therefore, these procedures were applied to samples from
211 the different treatments from one block only in the central area of the experimental site.

212 Soil respiration (Alef, 1995) was determined by measuring the CO_2 produced
213 during a 21-day incubation experiment at $28\pm 2^\circ\text{C}$, measured at 1, 7, 14 and 21 days
214 during the incubation period. For each treatment and the three replicates, 100 g of soil
215 adjusted to 65% of its water-holding capacity was placed in a 2 l hermetic glass jar with

216 a vial containing 25 ml 0.1M NaOH; the NaOH was titrated with 0.1M HCl with
217 phenolphthalein as the indicator.

218 *Physical soil analysis: methods for measuring aggregate stability*

219 Aggregate stability was measured first according to aggregate-size distribution (mean
220 weight diameter, MWD), which included three different methods for three different
221 initial disrupting forces: slow wetting (MWD_{SW}), fast wetting (MWD_{FW}) and stirring
222 after pre-wetting, which causes mechanical breakdown (MWD_{MB}). Water-stable
223 aggregates were also determined by the wet method (WSA_{WET}) and its modification
224 (WSA_{DRY}), which avoided initial slow pre-wetting. Four laboratory replicates were used
225 for each plot and every aggregate stability method.

226 The MWD relates to the aggregates that remain after the various treatments leading
227 to disaggregation. Treatments included: slow wetting which measures the stability of
228 aggregates affected by differential swelling (microcracking of aggregates), fast wetting
229 which measures the resistance of soil aggregates to the implosion caused by the
230 penetration of water into soil aggregates (slaking) and by the dissolution and dispersive
231 actions of water, and stirring after pre-wetting which measures the stability of
232 aggregates subjected to mechanical breakdown (stirring). Analyses were done following
233 the method of Amézqueta *et al.* (1996) which uses 1–2-mm diameter aggregates, but
234 includes the size fraction < 0.25 mm. These authors used ethanol in one of the tests and
235 in the measurement of disaggregation because it enables different breakdown
236 mechanisms to be studied independently. Ethanol prevents slaking and swelling because
237 of its relatively low surface tension and its much smaller dielectric charge than that of
238 water. In addition, particles do not reaggregate during drying (Merzouk & Blake, 1991).

239 The different disrupting mechanisms were applied to 4 g of soil aggregates: (i) slow
240 wetting, where aggregates were placed on a 0.25-mm mesh sieve and wetted until
241 saturation in a vapour chamber (deionized water), (ii) fast wetting, where aggregates
242 were placed on a 0.25-mm mesh sieve and immersed gently for 10 minutes in 100 ml of
243 deionized water and (iii) stirring after pre-wetting, where aggregates were immersed
244 gently in 50 ml of ethanol for 10 minutes. Following this, the ethanol was removed
245 carefully by decantation and the aggregates were transferred to an Erlenmeyer flask
246 filled with 50 ml deionized water, and the level was adjusted to 200 ml. The Erlenmeyer
247 was corked and agitated end over end 20 times, and left for 30 minutes to allow coarse
248 particles to settle. After removing the excess water by pipette, the remaining soil–water
249 mixture was transferred to a 0.25-mm mesh sieve.

250 In all procedures, the sieves were transferred to the modified Yoder apparatus and
251 disaggregation involved moving the sieves immersed in ethanol (95%) up and down
252 mechanically 10 times over a distance of 1.3 cm.

253 The fraction > 0.25 mm was oven-dried (105°C, 24hours) and dry-sieved for 1
254 minute on a column of four 6.5-cm diameter sieves with mesh sizes of 2.0, 1.0, 0.5 and
255 0.25 mm with a standard mechanical sieve shaker.

256 The aggregate stability for each treatment was expressed as MWD (μm). It was
257 calculated by Equation (1):

$$258 \quad \text{MWD} = \sum_{i=1}^n W_i \times D_i \times 10^3, \quad (1)$$

259 where n is the number of aggregate size fractions considered in the analysis, D_i is the
260 mean diameter of aggregates that can potentially stay in the i th and $i+1$ sieves in the
261 four size class: $D_1=1.5$ mm (≥ 1 mm to 2 mm), $D_2=0.75$ mm (≥ 0.5 mm to 1 mm),
262 $D_3=0.375$ mm (≥ 0.25 mm to 0.5 mm) and $D_4=0.125$ mm (< 0.25 mm), and W_i is the

263 mass percentage of each fraction, which equals the dry weight of aggregates in the i th
264 size fraction (g) divided by the sum of total sieved dry soil weight fractions (g)
265 including the smallest (< 0.25 mm) after disaggregation.

266 The standard test for water-stable aggregates (WSA_{WET}) is the single-sieve (0.25
267 mm) method (Kemper & Rosenau, 1986). We placed 4 g of a 1–2 mm air-dried sample
268 (Mt) on the sieve which was wetted with deionized water vapour (slow wetting) to
269 saturation. The sieve was placed in a modified Yoder apparatus and was raised and
270 lowered through a 1.3-cm vertical distance for 3 minutes at a frequency of 0.6 Hz in
271 deionized water. Soil remaining on the sieve was oven-dried (105°C, 24 hours) and
272 weighed to give the mass of the resistant or stable aggregates (Mr). After weighing,
273 aggregates were dispersed with an ultrasonic probe for 30 minutes. The fraction of each
274 sample remaining on the 0.25 mm sieve was oven-dried (105°C, 24 hours) and weighed
275 to obtain the mass of >0.25 mm sand (Ms). The mass percentage of water-stable
276 (macro)aggregates (WSA) was calculated by Equation (2):

$$277 \quad WSA (\%) = \frac{Mr - Ms}{Mt - Ms} \times 100 \quad (2)$$

278 In the modified test (WSA_{DRY}), air-dried samples (Wt) were processed directly
279 without being vapour wetted first in order to cause maximum disruption of aggregates.

280 *Statistical analysis*

281 The statistical analyses were done with SAS v9.4 (SAS Institute Inc., 2002–2012). Data
282 were analysed by an analysis of variance (ANOVA) and the residuals were checked for
283 normality and homogeneity of the variances. If these assumptions did not hold,
284 appropriate transformations were applied. The standard error of the difference between
285 means (SED) was computed and means were compared with Fisher's least significant
286 difference (LSD). Differences were considered significant at $P < 0.05$.

287 The samples (four for each treatment) used to measure respiration and aggregate
288 stability were taken from one block only, therefore, there is no true replication (pseudo-
289 replication) and any significant differences identified must be treated with caution. The
290 distances between samples within a plot can be larger than those between samples from
291 different treatments (plots are 7 to 11-m wide and 25-m long), and the results of these
292 properties are consistent with the differences obtained between treatments for the rest of
293 measurements which did have true replication (three blocks).

294 **Results**

295 *Aggregate stability*

296 The coefficient of variation (CV) of aggregate stability data, irrespective of the applied
297 test, was <19% except for WSA_{WET} for the 0–90SS treatment (CV=24%). The MWD_{MB} ,
298 MWD_{FW} and WSA_{DRY} tests enabled us to detect differences between fertilizer
299 treatments on aggregate stability (Table 2; Figure 2a,b). The WSA_{DRY} test established a
300 grading in the increase in stability following the treatment order (from the smallest to
301 the largest value): control → mineral → S12mo → S3mo. In slurry treatments, the
302 smallest values of MWD_{MB} were also recorded in treatments where it was applied 12
303 months before, however, the opposite occurred for MWD_{FW} (smallest values in S3mo
304 treatments). The gentle slow-wetting methods (WSA_{WET} , MWD_{SW}) reduced
305 disaggregation (Figure 2a,b), mainly in the larger (1–2 mm) aggregate class (MWD_{SW} ,
306 Figure 3).

307 *Exchangeable cations*

308 There were no significant differences between the three blocks in exchangeable cations.
309 Soil exchangeable Mg^{2+} (Figure 4) increased to $1.34 \text{ cmol}_c \text{ kg}^{-1}$ soil if slurries were

310 included in the fertilizer treatment compared to mineral fertilizer ($0.66 \text{ cmol}_c \text{ kg}^{-1} \text{ soil}$).
311 Otherwise, Na^+ exchangeable cations tended to increase with time in treatments that
312 received slurries, although the differences between fertilizer treatments were not
313 significant (Table 3; Figure 4). Differences in K^+ contents were variable and significant
314 differences depended upon which treatments were compared.

315 *Soil respiration*

316 Average soil respiration after 21 days of incubation (Tables 4, 5) in recent (three
317 months previously) PS-amended soil ($0.72\text{--}0.88 \text{ mg CO}_2\text{-C kg}^{-1} \text{ dry soil hour}^{-1}$) was
318 larger than from the control and mineral treatments ($0.41\text{--}0.48 \text{ mg CO}_2\text{-C kg}^{-1} \text{ dry soil}$
319 hour^{-1}) and also from treatments (S12mo, 0–60FS and 0–90SS), which had received
320 slurries at the smallest rate twelve months previously (0.64 and $0.47 \text{ mg CO}_2\text{-C kg}^{-1} \text{ dry}$
321 soil hour^{-1}). The main differences were more evident during the first 14 days of the
322 incubation period (Table 6; Figure 5).

323 Water stable aggregates (WSA_{DRY}) and mean weight diameter with fast wetting
324 (MWD_{FW}) were plotted against accumulated respiration in a 21-day period. As
325 accumulated respiration increased, the WSA_{DRY} increased but MWD_{FW} decreased
326 (Figure 6).

327 *Soil porosity*

328 The qualitative assessment of porosity by micromorphology showed that the soil treated
329 with mineral fertilizer and the control had mainly fissures and vughs, whereas samples
330 fertilized with slurry tended to have compound packing pores between aggregates; these
331 were more evident for treatment with slurry from fattening pigs (Figure 7). The image
332 analyses showed an overall increase of total porosity in the plots fertilized with slurry
333 (Figure 7), which is supported by the results of the analysis of variance (Tables 7, 8) of

334 total porosity ($> 25 \mu\text{m}$). The pore-size distribution showed that recent slurry
335 application at sowing (3 months before sampling) increased porosity in the ranges from
336 25 to 100 μm and 100 to 400 μm when compared with mineral fertilizer or with the
337 control (no nitrogen) plots (Table 8). These differences tend to disappear when slurry
338 had been applied twelve months before sampling (Tables 7, 8).

339 **Discussion**

340 The consistency of our results of aggregate stability is shown by the coefficients of
341 variation, which fall within the accepted ranges in the literature (Saygin *et al.* 2012).

342 The slow-wetting treatment (MWD_{SW} and WSA_{WET}) resulted in fewer differences
343 between fertilizer treatments (Table 2; Figure 2a,b). Slow-wetting before wet-sieving of
344 aggregates is an accepted practice because maximum aggregate stability is obtained in
345 saturated samples (Sun *et al.*, 1995). If later sieving is done in ethanol, the intensity of
346 disaggregation is even more limited (Le Bissonnais, 1996). We observed this in the
347 larger MWD of aggregates subjected to the slow-wetting procedure (Figure 3a,b,c).

348 An increase in Mg^{2+} , which occurred with our slurry treatments (Figure 4), might
349 cause considerable swelling if expanding clays are present. In our case they were absent
350 because illite and chlorite are the dominant clays (Figure 1). Because no significant
351 variation was detected in exchangeable Na^+ (Table 3; Figure 4), the observed changes in
352 aggregate stability for the different methods cannot be attributed to changes in the type
353 of adsorbed cations or to increments in the sodium adsorption ratio (SAR).

354 Our slurries had a small C:N ratio ($\sim 6\text{--}8$, Table 1) with mineral $\text{NH}_4^+\text{-N}$ up to 70%
355 of total N (Table 1). The large initial availability of N when slurries had been applied
356 just after stubble incorporation might reduce the proportion of macroaggregates
357 (Bossuyt *et al.*, 2001). The small MWD_{FW} in S3mo samples (Figure 2a) because of the

358 lack of macroaggregates (Figure 3a) supports this hypothesis. In contrast, the WSA_{DRY}
359 test indicates a better resistance to slaking disaggregation in S3mo samples than in the
360 0–120M treatment (where no mineral N had been applied until tillering of the cereal)
361 with the larger number of macroaggregates ($> 250 \mu\text{m}$). In fresh PS, organic carbon
362 content is mainly in a water-soluble form (Bernal *et al.*, 1991) and hemicellulose is the
363 major polysaccharide (Müller, 1980) with a content that might be up to 89% greater
364 than that of cellulose, although lignin and cellulose are almost undigested by pigs.
365 Polysaccharides can act as transitory binding agents (Tisdall & Oades, 1982). The
366 predominance of easily degradable compounds is corroborated by soil respiration after
367 21 days of incubation in recent (S3mo) PS-amended soil (Tables 4, 5). The relation
368 between WSA_{DRY} and accumulated respiration (Figure 6) indicates more microbial
369 activity occurs when the soil OM light fraction is greater (Yagüe *et al.*, 2012b). By
370 opposite, when MWD_{FW} is introduced as the stability test (Figure 6), the relation means
371 that some transient binding agents (glues) associated with recent slurry incorporation
372 (30FS treatments at sowing) could be affected first by dissolution with 10 minutes of
373 immersion in deionized water, and second by ethanol because polysaccharides are
374 soluble in both liquids (Lawther *et al.*, 1995). These transient binding agents are easily
375 degraded by microorganisms, which contribute to the relatively large rates of respiration
376 and lead to a negative relation with MWD_{FW} . Solubilization was reduced as the soil OM
377 became more protected with time.

378 The largest values of MWD_{FW} in S12mo samples can be explained by the nature of
379 the soil. In carbonate-rich soil, aggregate formation associated with microbial activity
380 co-exists with physicochemical pathways of aggregate formation. Fernández-Ugalde *et*
381 *al.* (2011) hypothesized that macroaggregate stabilization mainly results from the
382 formation of physical coatings and pore infillings of fine-sized secondary carbonates

383 that impregnate the light fraction of OM and decrease the porosity inside the aggregates,
384 which slows down the turnover of OM. This interaction between carbonates and added
385 organic matter possibly explains the larger MWD_{FW} values where slurry had been
386 applied at cereal tillering only, twelve months before sampling (Figure 2a).

387 Differences between S12mo and S3mo also resulted in the MWD_{MB} test (Figure 2a),
388 but they show the reverse trend to that in MWD_{FW} , probably because carbonate coatings
389 do not protect the aggregates effectively from stirring. In our case, the mechanical
390 energy applied in the MWD_{MB} test broke up the aggregates in the upper size range (1–2
391 mm), but it was insufficient to break up aggregates of the next size down; the main
392 proportion of aggregates was in the 0.5–1 mm range (Figure 3c).

393 The effect of PS application on aggregate stability to mitigate against slaking
394 (MWD_{FW} or WSA_{DRY} values) is important because slaking occurs when aggregates are
395 wetted rapidly. Rapid wetting occurs in our Mediterranean climate because the soil
396 surface is usually dry when the erratic rainfall occurs, and there is also little soil cover
397 from sowing until tillering. Furthermore, non-expanding clays such as illite, chlorite and
398 to a lesser extent kaolinite predominate in our soil and are more likely to be associated
399 with slaking than swelling clays (Emerson, 1964). In addition, the clay content of our
400 soil is small, which also means that slaking is more likely (Le Bissonnais, 1996).

401 Although aggregate stability does not necessarily decrease with decreasing
402 macroporosity (Bresson & Moran, 2004), the decreasing stability in WSA_{DRY} (Figure
403 2b) is followed by a decrease of macroporosity in the 25–200 μm range.

404 Furthermore, total porosity ($>25 \mu\text{m}$) under mineral fertilizer did not differ
405 significantly (Table 8) from that under slurry fertilizer. This could arise from the
406 development of fissures (planar voids) that masked useful porosity (e.g. 0–120M,
407 Figure 7). The differences that appear at the 25–100 μm pore size (at which the S3mo

408 treatments surpassed mineral treatment) are important because these pores are packing
409 pores between the granular aggregates, and as such are involved in capillary movement
410 of water and soil aeration (Oades, 1984). These differences also appear, although less
411 clearly, between the 100 and 400 μm pore sizes, which correspond to interaggregate or
412 faunal pores involved in the fast drainage of water and root growth. As differences
413 disappear with time (mineral and S12mo), they could favour water infiltration in the
414 early stages of crop growth, which coincides with winter rains here. This idea requires
415 further research.

416 Overall differences between more recent to older slurry treatments, or between
417 more recent slurry and mineral fertilizer treatments are in accord with Du *et al.* (2016)
418 who found an increase in aggregates between 0.25–0.5 mm after the application of
419 biogas pig slurry. They suggested that the controlling factor for the formation of these
420 aggregates and microaggregates resulted from the combination of high specific surface
421 area of the organic molecules in the slurry together with the action of the functional
422 groups of the slurry hydrophilic colloids (Liang *et al.*, 2005). Similarly, Grunwald *et al.*
423 (2016) explained this effect by the more fluid nature of slurry than biochar or manure as
424 aggregants, allowing it to spread more readily over the surfaces of soil components and
425 enhanced their ability to form aggregates.

426 **Conclusions**

427 In this dryland system, slaking is the main agent for aggregate breakdown in calcareous
428 soil. Nevertheless, if PS is the fertilizer, the stability tests (MWD_{FW} and WSA_{DRY}) can
429 result in contradictory assessments on resistance to slaking. When WSA_{DRY} reached the
430 largest values, MWD_{FW} values were the smallest probably because of the water's
431 interaction with transient binding agents (water-soluble glues) from PS. Nevertheless,

432 further research is needed on the nature and evolution over time of these binding agents.
433 Carbonates probably prevent OM decomposition, which can be deduced from evolution
434 of the rate of respiration during a growing season and from differences in aggregate
435 stability when mineral and organic fertilizers were compared. The introduction of pig
436 slurry into fertilization strategies benefits porosity, mainly in the 25–200 μm range area,
437 but again this is a transient effect. These pores are associated with the capillary
438 movement of water, soil aeration, fast drainage of water and root growth, therefore this
439 could be important in the water dynamics of the early crop cycle.

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558 **FIGURE CAPTIONS**

559 **Figure 1** Diffraction spectra (X-rays) of raw, glycolated (for 48 hours) and heated soil
560 samples. The clay phases identified are included.

561 **Figure 2** Average values of stability of aggregate tests: (a) mean weight diameter
562 (MWD), slow wetting (SW), fast wetting (FW) and stirring after pre-wetting
563 (mechanical breakdown, MB) and (b) water stable aggregates (WSA) standard
564 method by Kemper & Rosenau (1986) with pre-wetting (WET) and the modified
565 method without pre-wetting (DRY) for each fertilizer treatment: mineral (Mineral),
566 slurry applied twelve months before sampling (S12mo) or slurry applied three
567 months before sampling (S3mo). Treatment acronyms are described in Table 1.

568 **Figure 3** Size distribution of aggregates after fractionation into four classes (> 1 to 2
569 mm, > 0.50 to 1 mm, > 0.25 to 0.50 mm and < 0.25 mm) following three methods:
570 (a) fast wetting, (b) slow wetting and (c) stirring after pre-wetting (mechanical
571 breakdown), and for different fertilizer treatments: mineral (Mineral), slurry
572 applied twelve months before sampling (S12mo) or slurry applied three months
573 before sampling (S3mo). Treatment acronyms are described in Table 1.

574 **Figure 4** Average values of exchangeable cations (K^+ , Mg^{2+} , Na^+ and Ca^{2+}) for each
575 fertilizer treatment: mineral (Mineral), slurry applied twelve months before
576 sampling (S12mo) or slurry applied three months before sampling (S3mo).
577 Treatment acronyms are described in Table 1.

578 **Figure 5** Soil respiration average ($mg\ CO_2-C\ kg^{-1}\ soil\ hour^{-1}$) after 1, 7, 14 and 21 days
579 of incubation for each treatment: mineral fertilizer (Mineral), slurry applied twelve
580 months before sampling (S12mo) or slurry applied three months before sampling
581 (S3mo). Treatment acronyms are described in Table 1.

582 **Figure 6** Water aggregate stability modified without pre-wetting ($WSA_{DRY, \bullet}$) and mean
583 weight diameter after fast wetting ($MWD_{FW, \blacksquare}$) plotted against total cumulative
584 respiration ($\text{mg CO}_2\text{-C kg}^{-1}$ dry soil) for 21 days of incubation. The coefficient of
585 determination (R^2) shows the strength of association between the variables.

586 **Figure 7** Images of soil porosity (in black) from different fertilizer treatments: 0–0, 0–
587 120M, 30FS–0; 30FS–60M, 30FS–20PS and 30FS–60SS. Size of each image is
588 31.5-mm wide and 42.0-mm long. Acronyms of treatments are described in Table
589 1.

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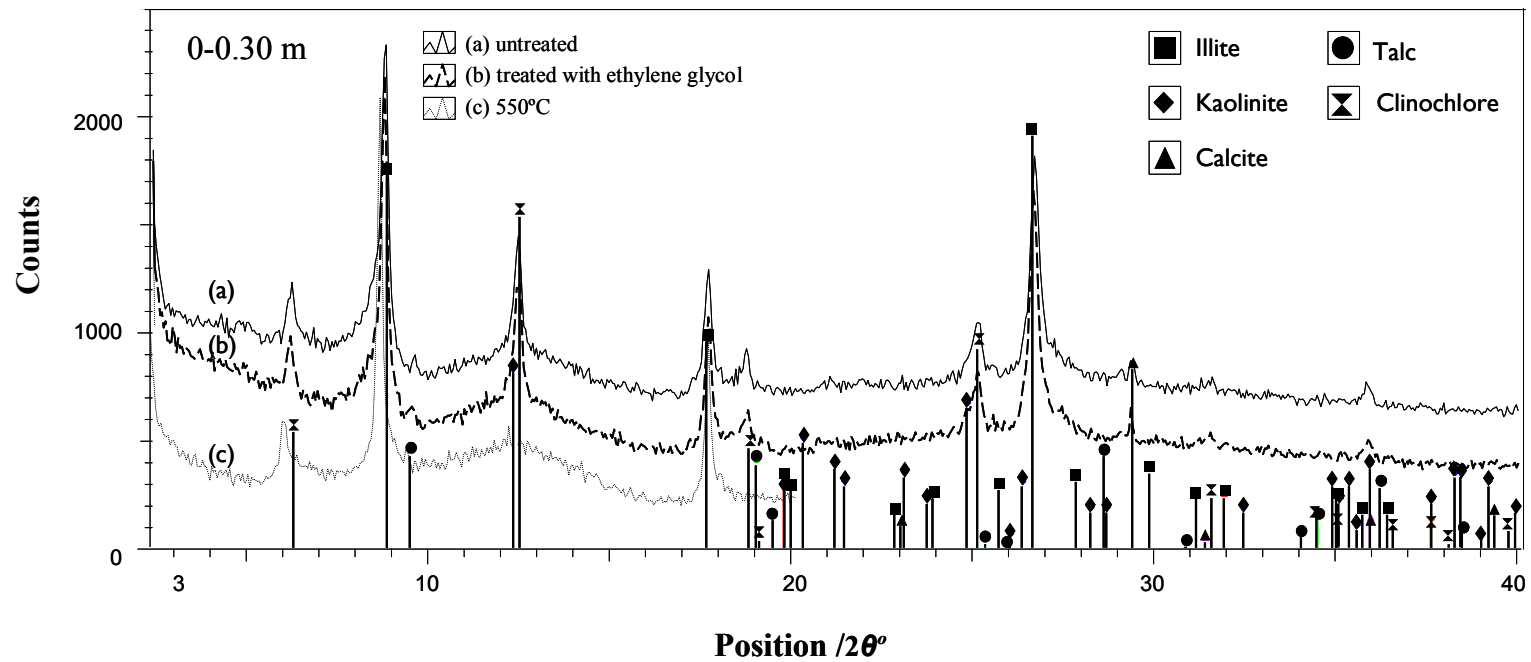


Figure 1 Diffraction spectra (X-rays) of raw, glycolated (for 48 hours) and heated soil samples. The clay phases identified are included.

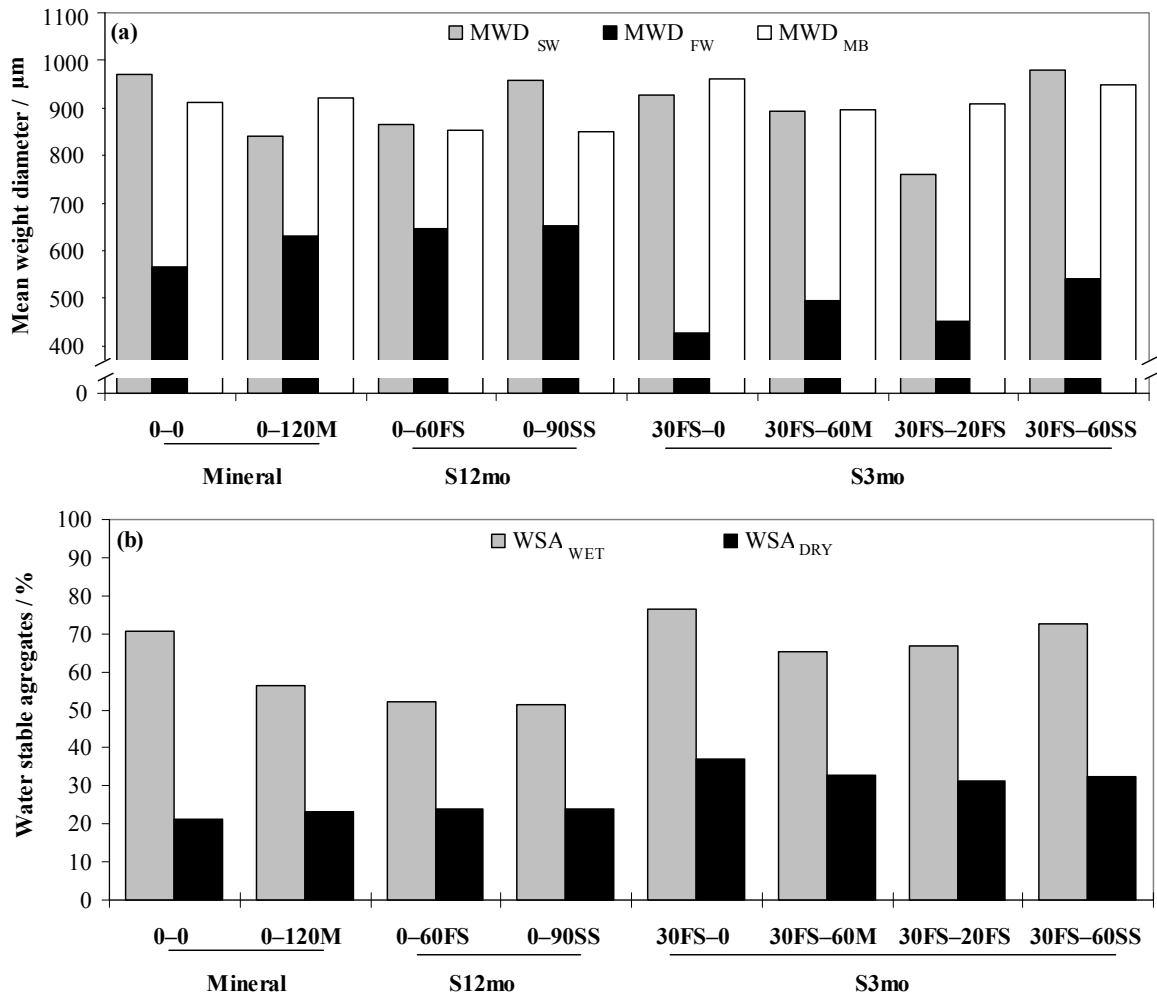


Figure 2 Average values of stability of aggregate tests: (a) mean weight diameter (MWD), slow wetting (SW), fast wetting (FW) and stirring after pre-wetting (mechanical breakdown, MB) and (b) water stable aggregates (WSA) standard method by Kemper & Rosenau (1986) with pre-wetting (WET) and the modified method without pre-wetting (DRY) for each fertilizer treatment: mineral (Mineral), slurry applied twelve months before sampling (S12mo) or slurry applied three months before sampling (S3mo). Treatment acronyms are described in Table 1.

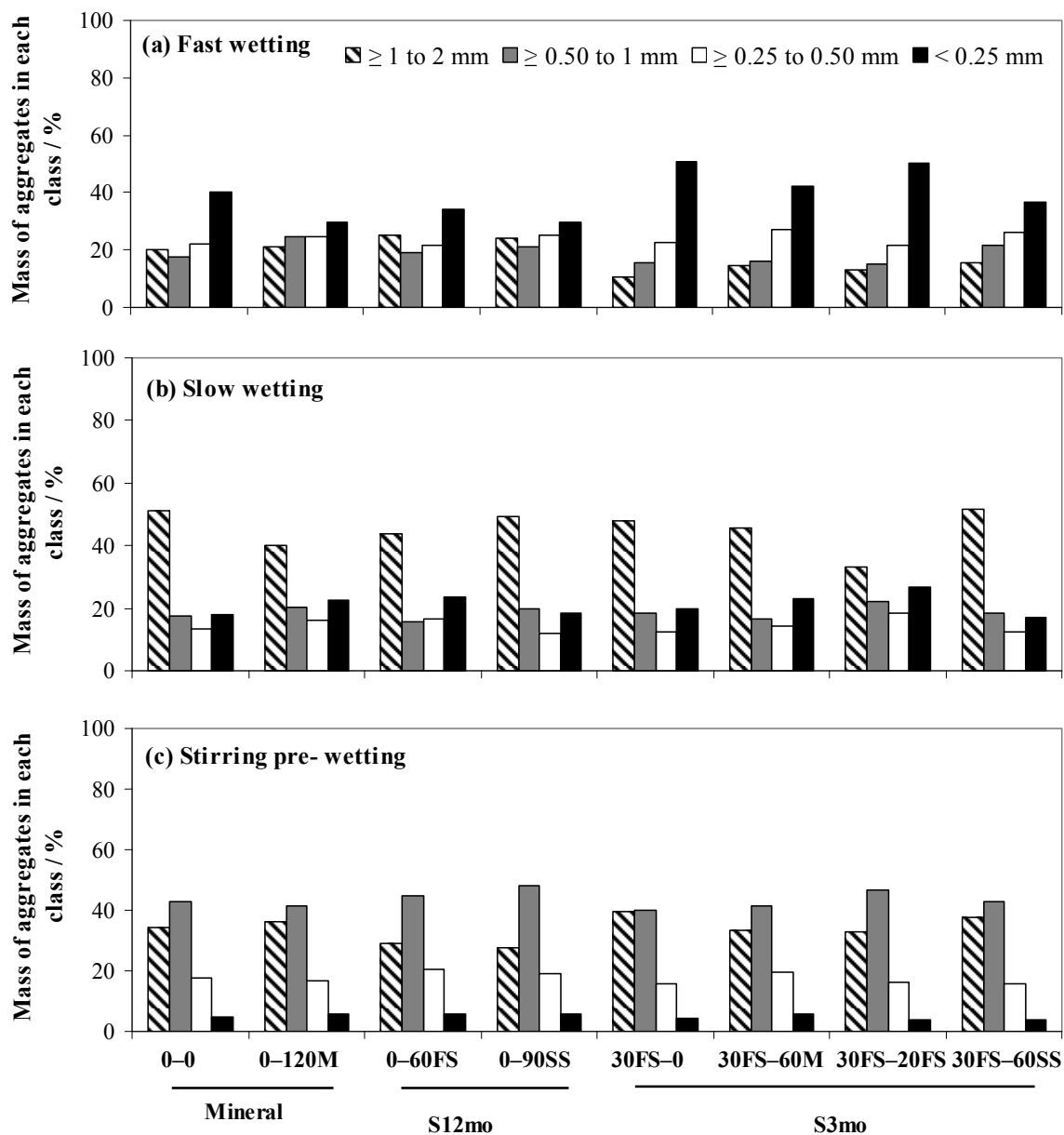


Figure 3 Size distribution of aggregates after fractionation into four classes (> 1 to 2 mm, > 0.50 to 1 mm, > 0.25 to 0.50 mm and < 0.25 mm) following three methods: (a) fast wetting, (b) slow wetting and (c) stirring after pre-wetting (mechanical breakdown), and for different fertilizer treatments: mineral (Mineral), slurry applied twelve months before sampling (S12mo) or slurry applied three months before sampling (S3mo). Treatment acronyms are described in Table 1.

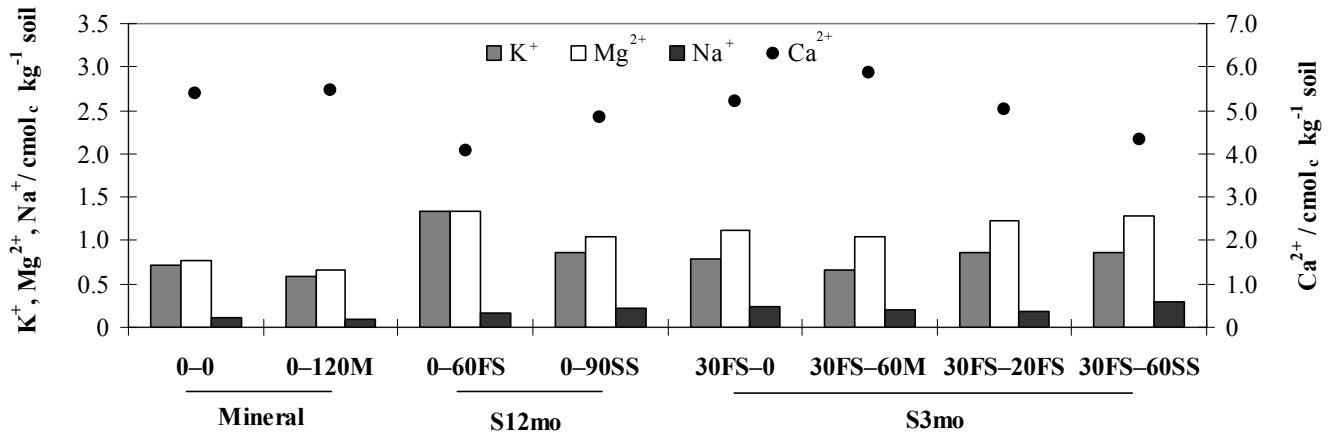


Figure 4 Average values of exchangeable cations (K⁺, Mg²⁺, Na⁺ and Ca²⁺) for each fertilizer treatment: mineral (Mineral), slurry applied twelve months before sampling (S12mo) or slurry applied three months before sampling (S3mo). Treatment acronyms are described in Table 1.

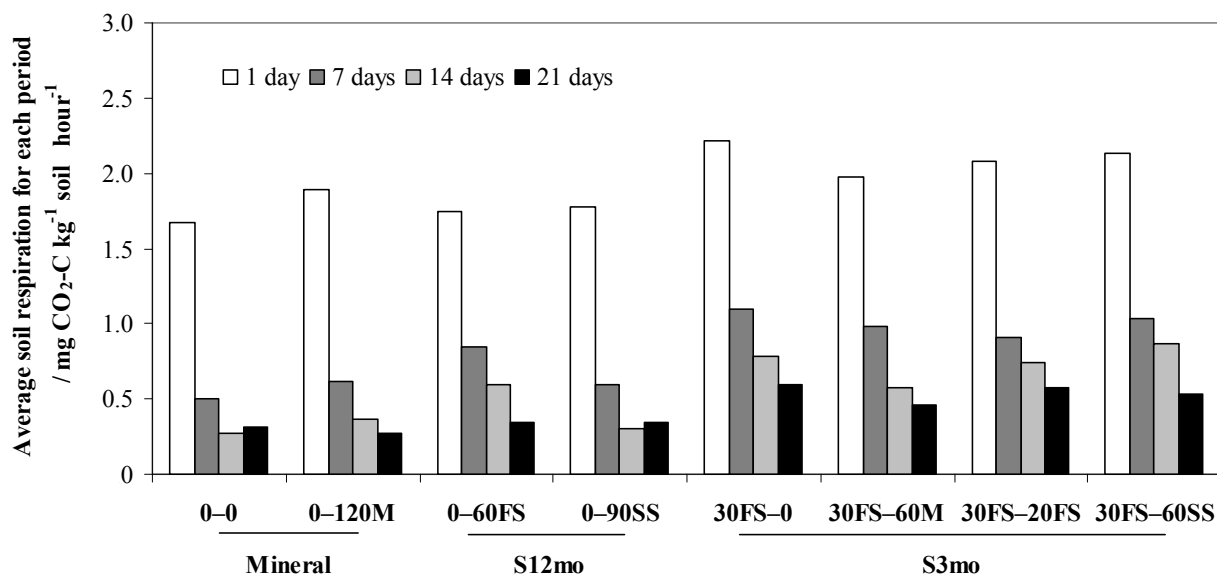


Figure 5 Soil respiration average ($\text{mg CO}_2\text{-C kg}^{-1} \text{ soil hour}^{-1}$) after 1, 7, 14 and 21 days of incubation for each treatment: mineral fertilizer (Mineral), slurry applied twelve months before sampling (S12mo) or slurry applied three months before sampling (S3mo). Treatment acronyms are described in Table 1.

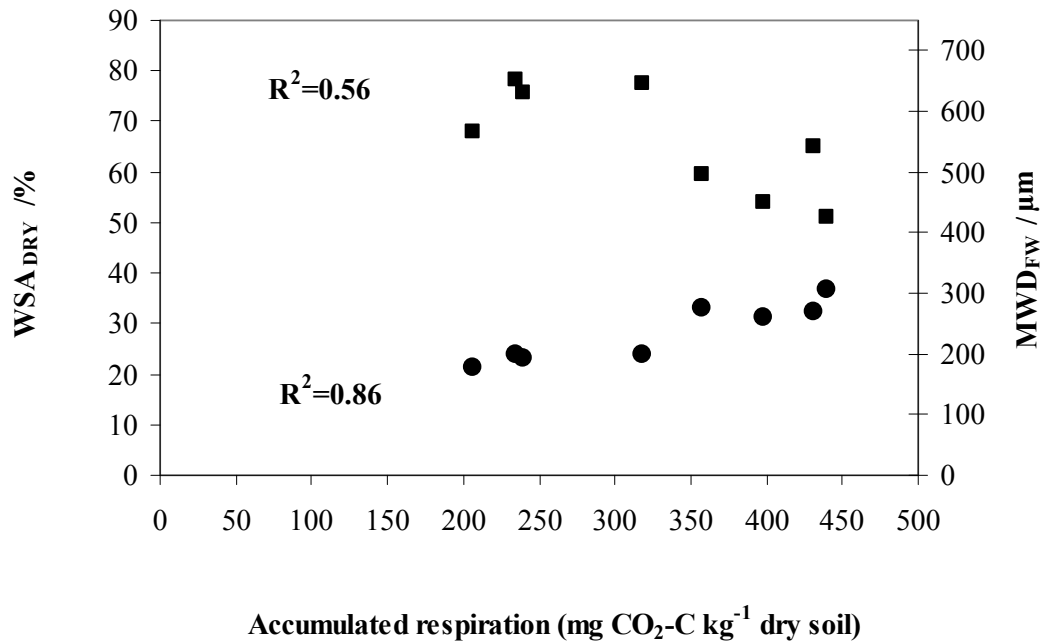


Figure 6 Water aggregate stability modified without pre-wetting (WSA_{DRY}, ●) and mean weight diameter after fast wetting (MWD_{FW}, ■) plotted against total cumulative respiration (mg CO₂-C kg⁻¹ dry soil) for 21 days of incubation. The coefficient of determination (R^2) shows the strength of association between the variables.

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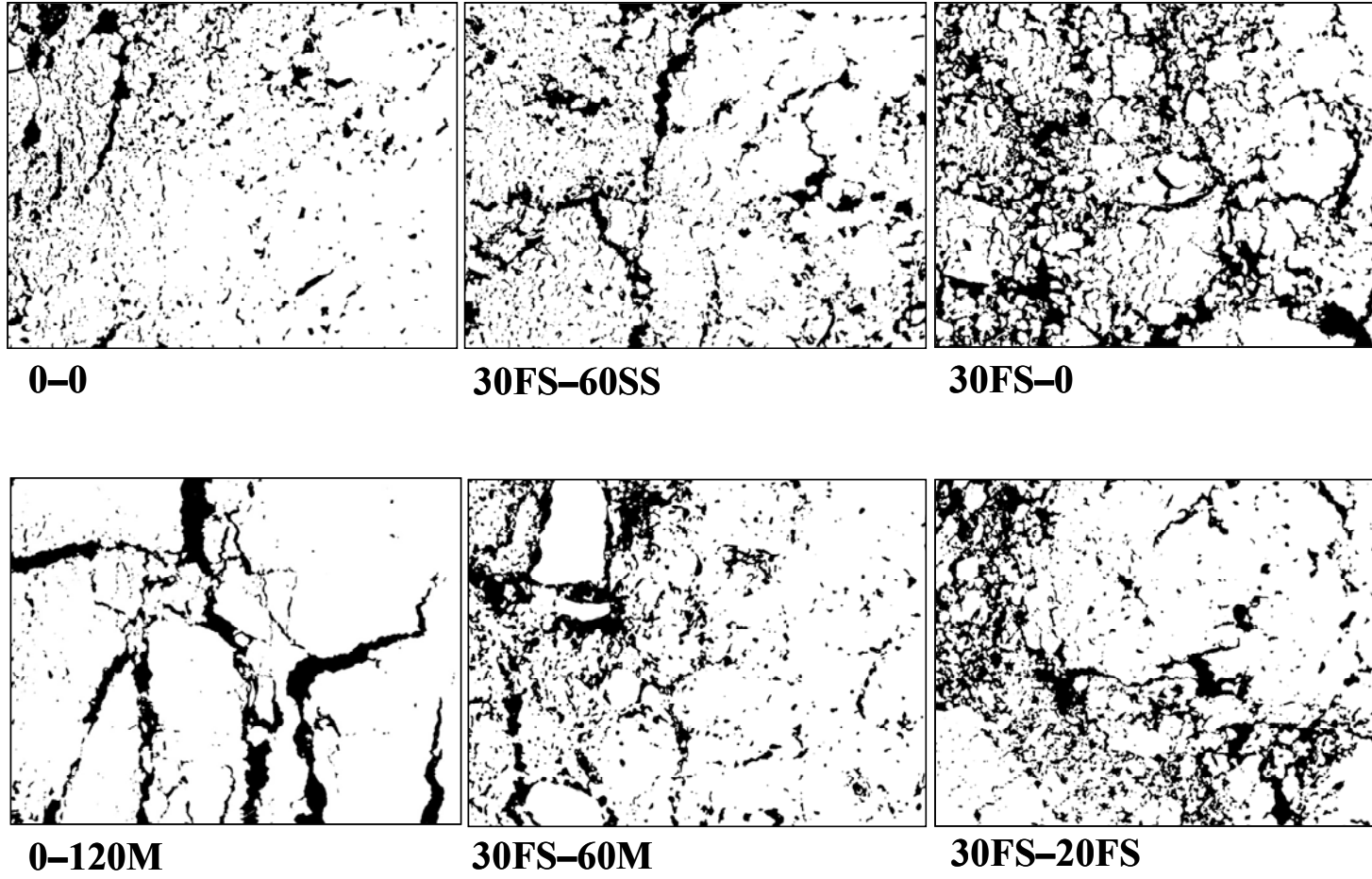


Figure 7 Images of soil porosity (in black) from different fertilizer treatments: 0-0, 0-120M, 30FS-0; 30FS-60M, 30FS-20PS and 30FS-60SS. Size of each image is 31.5-mm wide and 42.0-mm long. Acronyms of treatments are described in Table 1.

625 **Table 1.** Annual fertilizer treatments including averages of the total nitrogen (TN), organic N (Norg) and organic matter (OM) applied and
 626 accumulated (seven cereal growing seasons) grain yields (0% humidity). Fertilizers (mineral or from different slurry sources) were applied
 627 annually at presowing (PreS) or tillering as a top-dressing (TopD) or both
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Sampling time and last fertilizer application ^a	Treatment	Annual fertilizer split		2002 to 2010 (7 campaigns)			Presowing fertilization in 2010– 2011 campaign			Accumulated grain yield /kg ha ⁻¹
		PreS	TopD	TN	Norg /kg ha ⁻¹ year ⁻¹ (SD ^e)	OM	TN	Norg /kg ha ⁻¹ year ⁻¹ (SD ^e)	OM	
12-mo, mineral	0–0	0	0	0	0	0	0	0	0	18 373
	0–120M ^b	0	120M	120	0	0	30	0	0	21 550
12-mo, slurry (S12mo)	0–60FS ^c	0	60FS	405 (76)	130 (70)	2 622 (784)	0	0	0	20 897
	0–90SS ^d	0	90SS	175 (102)	61 (48)	1 764 (1 228)	0	0	0	24 800
3-mo, slurry (S3mo)	30FS–0	30FS	0	194 (48)	59 (15)	1 681 (240)	170 (1)	64 (2)	1 854 (10)	21 153
	30FS–60M	30FS	60M	254 (48)	59 (15)	1 681 (240)	170 (1)	64 (2)	1 854 (10)	23 640
	30FS–20FS	30FS	20FS	320 (33)	102 (25)	2 491 (254)	170 (1)	64 (2)	1 854 (10)	22 363
	30FS–60SS	30FS	60SS	279 (44)	82 (17)	2 107 (273)	170 (1)	64 (2)	1 854 (10)	22 329

629 ^a12-mo and 3-mo, the last fertilizer was applied twelve or three months before sampling, respectively.

630 ^bM, mineral fertilizer applied as ammonium nitrate (AN). Numbers indicate the rate applied: 60 kg N ha⁻¹ year⁻¹ or 120 kg N ha⁻¹ year⁻¹.

631 ^cFS, slurry from fattening pigs. Numbers indicate the average theoretical applied rate: 20 t ha⁻¹ year⁻¹, 30 t ha⁻¹ year⁻¹ or 60 t ha⁻¹ year⁻¹.

632 ^dSS, slurry from sows. Numbers indicate the average theoretical applied rate: 60 t ha⁻¹ year⁻¹ or 90 t ha⁻¹ year⁻¹.

633 ^eSD, standard deviation.

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Table 2. Analysis of variance (four replicates) of aggregate stability (Ag-Stab) with different tests: (a) mean weight diameter (MWD / μm), slow wetting (SW), fast wetting (FW) and stirring after pre-wetting (mechanical breakdown, MB) and (b) water stable aggregates (WSA /%) standard method by Kemper & Rosenau (1986) with pre-wetting (WET) and modified method without pre-wetting (DRY) for the different fertilizer treatments

Ag-Stab test	Source	Degrees of freedom	Sum of squares	Mean square	<i>F</i> ratio	<i>P</i>
MWD _{SW}	Between treatments	7	155 967.9	22 281.1	0.72	0.65
	Within treatments (residual)	24	741 671.4	30 903.0		
	Total	31	897 639.3			
	SE ^a	101.49				
MWD _{FW}	Between treatments	7	215 818.0	30 831.1	11.21	<0.0001
	Within treatments (residual)	24	65 980.8	2 749.2		
	Total	31	281 798.8			
	SE, SED, LSD ^a	30.27, 37.08, 76.52				
MWD _{MB}	Between treatments	7	43 712.7	6 244.7	9.71	<0.0001
	Within treatments (residual)	24	15 436.0	643.2		
	Total	31	59 148.7			
	SE, SED, LSD ^a	14.64, 17.93, 37.01				
WSA _{WET}	Between treatments	7	2 716.2	388.0	1.17	0.36
	Within treatments (residual)	24	7 987.2	332.8		
	Total	31	10 703.4			
	SE ^a	10.53				
WSA _{DRY}	Between treatments	7	861.6	123.1	3.90	0.006
	Within treatments (residual)	24	757.4	31.6		
	Total	31	1 619.0			
	SE, SED, LSD ^a	3.24, 3.97, 8.20				

^aSE, standard error of the mean /%; SED, standard error of a difference /%; LSD, least significant difference test /%; all for *P*= 0.05 with 24 degrees of freedom.

Table 3. Analysis of variance of exchangeable cations (K^+ , Mg^{2+} , Na^+ and Ca^{2+}) expressed in $cmol_c\ kg^{-1}$ soil (three replicates) for the different fertilizer treatments

Exchangeable cations	Source	Degrees of freedom	Sum of squares	Mean square	<i>F</i> ratio	<i>P</i>
K^+	Between treatments	7	1.054	0.150	23.19	<0.0001
	Between blocks	2	0.004	0.002	0.28	0.76
	Within treatments (residual)	14	0.091	0.006		
	Total	23	1.149			
SE, SED, LSD ^a		0.046, 0.066, 1.141				
Mg^{2+}	Between treatments	7	1.231	0.176	5.35	0.0038
	Between blocks	2	0.009	0.005	0.14	0.87
	Within treatments (residual)	14	0.460	0.033		
	Total	23	1.700			
SE, SED, LSD ^a		0.105, 0.148, 0.317				
Na^+	Between treatments	7	0.098	0.014	1.89	0.15
	Between blocks	2	0.017	0.008	1.13	0.35
	Within treatments (residual)	14	0.104	0.007		
	Total	23	0.218			
SE ^a		0.050				
Ca^{2+}	Between treatments	7	7.511	1.073	0.94	0.50
	Between blocks	2	1.184	0.592	0.52	0.60
	Within treatments (residual)	14	15.905	1.136		
	Total	23	24.600			
SE ^a		0.615				

^aSE, standard error of the mean /%; SED, standard error of a difference /%; LSD, least significant difference test /%; all for *P*= 0.05 with 14 degrees of freedom.

Table 4. Analysis of variance of average hourly soil respiration (mg CO₂-C kg⁻¹ dry soil hour⁻¹) and accumulated respiration (mg CO₂-C kg⁻¹ dry soil) after 21 days of incubation (three replicates) for the different fertilizer treatments

Variable	Source	Degrees of freedom	Sum of squares	Mean square	<i>F</i> ratio	<i>P</i>
Average respiration	Between treatments	7	0.724	0.104	23.36	<0.0001
	Within treatments (residual)	16	0.071	0.004		
	Total	23	0.797			
Accumulated respiration	Between treatments	7	181 093	25 870	23.36	<0.0001
	Within treatments (residual)	16	17 475	1 092		
	Total	23	198 568			

Table 5. Average hourly soil respiration and accumulated respiration after 21 days of incubation (three replicates) obtained for each methodology and fertilizer treatment

Sampling time and last fertilizer application ^a	Treatment	Average /mg CO ₂ -C kg ⁻¹ dry soil hour ⁻¹	Accumulated /mg CO ₂ -C kg ⁻¹ dry soil
12-mo, mineral	0-0	0.41	207
	0-120M ^b	0.48	240
12-mo, slurry (S12mo)	0-60FS ^c	0.64	318
	0-90SS ^d	0.47	234
3-mo, slurry (S3mo)	30FS-0	0.88	440
	30FS-60M	0.72	358
	30FS-20FS	0.82	398
	30FS-60SS	0.87	431
SE, SED, LSD ^e		0.0365, 0.0545, 0.1154	19.08, 26.98, 57.20

^a12-mo, 3-mo, the last fertilizer was applied twelve or three months before sampling, respectively.

^bM, mineral fertilizer applied as ammonium nitrate (AN). Numbers indicate the rate applied: 60 kg N ha⁻¹ year⁻¹ or 120 kg N ha⁻¹ year⁻¹.

^cFS, slurry from fattening pigs. Numbers indicate the average theoretical applied rate: 20 t ha⁻¹ year⁻¹, 30 t ha⁻¹ year⁻¹ or 60 t ha⁻¹ year⁻¹.

^dSS, slurry from sows. Numbers indicate the average theoretical applied rate: 60 t ha⁻¹ year⁻¹ or 90 t ha⁻¹ year⁻¹.

^eSE, standard error of the mean /%; SED, standard error of a difference /%; LSD, least significant difference test /%; all for $P= 0.05$ with 16 degrees of freedom.

Table 6. Analysis of variance of soil respiration average (mg CO₂-C kg⁻¹ soil hour⁻¹) after 1, 7, 14, and 21 days of incubation (three replicates) for different fertilizer treatments

Incubation period	Source	Degrees of freedom	Sum of squares	Mean square	F ratio	P
1 day	Between treatments	7	0.845	0.121	1.17	0.37
	Within treatments (residual)	16	1.650	0.103		
	Total	23	2.495			
	SE ^a	0.185				
7 days	Between treatments	7	1.066	0.152	19.59	<0.0001
	Within treatments (residual)	16	0.124	0.008		
	Total	23	1.191			
	SE, SED, LSD ^a	0.051, 0.072, 0.153				
14 days	Between treatments	7	1.115	0.159	16.34	<0.0001
	Within treatments (residual)	16	0.156	0.010		
	Total	23	1.271			
	SE, SED, LSD ^a	0.057, 0.081, 0.171				
21 days	Between treatments	7	0.346	0.049	4.60	0.0055
	Within treatments (residual)	16	0.172	0.011		
	Total	23	0.518			
	SE, SED, LSD ^a	0.061, 0.085, 0.180				

^aSE, standard error of the mean /%; SED, standard error of a difference /%; LSD, least significant difference test /%; all for $P= 0.05$ with 16 degrees of freedom.

Table 7. Analysis of variance of pore apparent diameter ranges according to different fertilizer treatments

Range	Source	Degrees of freedom	Sum of squares	Mean square	F ratio	P
> 25 μm	Between treatments	7	11.620	1.660	3.16	0.0082
	Between blocks	2	8.973	4.486	8.55	0.0007
	Between samples within treatments	16	11.464	0.717	1.37	0.2015
	Within samples (residual)	46	24.142	0.525		
	Total	71	56.199			
25–65 μm	Between treatments	7	0.680	0.097	3.74	0.0028
	Between blocks	2	0.504	0.252	9.70	0.0003
	Between samples within treatments	16	0.436	0.027	1.05	0.4269
	Within samples (residual)	46	1.195	0.026		
	Total	71	2.815			
65–100 μm	Between treatments	7	0.150	0.214	3.78	0.0026
	Between blocks	2	0.415	0.208	3.66	0.0336
	Between samples within treatments	16	0.543	0.034	0.60	0.8691
	Within samples (residual)	46	2.610	0.057		
	Total	71	5.068			
100–200 μm	Between treatments	7	5.445	0.778	5.42	0.0001
	Between blocks	2	0.979	0.489	3.41	0.0416
	Between samples within treatments	16	1.172	0.073	0.51	0.9284
	Within samples (residual)	46	6.600	0.143		
	Total	71	14.195			
200–400 μm	Between treatments	7	7.973	1.139	5.46	0.0001
	Between blocks	2	3.891	1.946	9.33	0.0004
	Between samples within treatments	16	2.903	0.181	0.87	0.6053
	Within samples (residual)	46	9.597	0.209		
	Total	71	24.364			
>400 μm	Between treatments	7	5.558	0.794	1.08	0.3930
	Between blocks	2	9.270	4.635	6.29	0.0038
	Between samples within treatments	16	15.445	0.965	1.31	0.2321
	Within samples (residual)	46	33.900	0.737		
	Total	71	64.170			

The porosity data /% for each interval were transformed ($X^{0.5}$) to fulfil the requirements of normality and homogeneity of variances in ANOVA analysis.

Table 8. Porosity^a areas for each fertilizer treatment and with different apparent pore diameter ranges

Sampling time and last fertilizer application ^b	Treatment	Total porosity /%			Pore ranges /%		
		>25 μm	25–65μm	65–100μm	100–200μm	200–400μm	>400μm
12-mo, mineral	0–0	12.0 (3.37)	0.5 (0.66)	1.3 (1.12)	2.7 (1.63)	2.6 (1.58)	4.8 (1.98)
	0–120M ^c	17.3 (4.01)	0.3 (0.57)	1.3 (1.12)	3.6 (1.84)	4.6 (2.05)	7.4 (2.49)
12-mo, Slurry (S12mo)	0–60FS ^d	19.8 (4.35)	0.5 (0.67)	1.7 (1.26)	4.1 (1.97)	5.0 (2.18)	8.5 (2.70)
	0–90SS ^e	15.2 (3.88)	0.7 (0.80)	1.8 (1.31)	3.7 (1.89)	3.8 (1.92)	5.3 (2.10)
3-mo, Slurry (S3mo)	30FS–0	23.2 (4.75)	0.8 (0.86)	2.5 (1.56)	6.5 (2.54)	8.0 (2.76)	5.3 (2.17)
	30FS–60M	17.6 (4.16)	0.6 (0.78)	1.9 (1.38)	4.5 (2.10)	5.1 (2.22)	5.5 (2.26)
	30FS–20FS	20.6 (4.46)	0.8 (0.86)	2.2 (1.47)	5.8 (2.38)	6.5 (2.50)	5.2 (2.13)
	30FS–60SS	15.1 (3.82)	0.6 (0.79)	1.8 (1.34)	4.2 (2.03)	4.6 (2.10)	3.8 (1.73)
SE, SED, LSD ^f		0.42, 0.34, 0.69	0.10, 0.08, 0.15	0.13, 0.11, 0.23	0.22, 0.18, 0.36	0.26, 0.22, 0.43	0.50, -, -

^aNumbers in brackets are the transformed values $x^{0.5}$.

^b12-mo and 3-mo, the last fertilizer was applied twelve months or three months before sampling, respectively.

^cM, mineral fertilizer applied as ammonium nitrate (AN). Numbers indicate the rate applied: 60 kg N ha⁻¹ year⁻¹ or 120 kg N ha⁻¹ year⁻¹.

^dFS, slurry from fattening pigs. Numbers indicate the average theoretical applied rate: 20 t ha⁻¹ year⁻¹, 30 t ha⁻¹ year⁻¹ or 60 t ha⁻¹ year⁻¹.

^eSS, slurry from sows. Numbers indicate the average theoretical applied rate: 60 t ha⁻¹ year⁻¹ or 90 t ha⁻¹ year⁻¹.

^fSE, standard error of the mean /%; SED, standard error of a difference /%; LSD, least significant difference test /%; all for $P= 0.05$ with 46 degrees of freedom.