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### **► To cite this version:**

Julie Jimenez, Marco Grigatti, Elisa Boanini, Dominique Patureau, Nicolas Bernet. The impact of biogas digestate typology on nutrient recovery for plant growth: Accessibility indicators for first fertilization prediction. *Waste Management*, 2020, 117, pp.18-31. <10.1016/j.wasman.2020.07.052>. <hal-02921497>

**HAL Id: hal-02921497**

**<https://hal.inrae.fr/hal-02921497v1>**

Submitted on 22 Aug 2022

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1 **The impact of biogas digestate typology on nutrient recovery for plant growth: accessibility**  
2 **indicators for first fertilization prediction**

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10 **Abstract**

11 In recent years, anaerobic digestion of organic waste (OW) is rapidly appearing as a winning waste  
12 management strategy by producing energy and anaerobic digestates that can be used as fertilizers in  
13 agricultural soils. In this context, the management of the OW treatment process to maximize agro-  
14 system sustainability satisfying the crop nutrient demands represents the main goal. To investigate  
15 these traits, two protocols to assess the plant availability of digestate nitrogen (N) and phosphorus (P)  
16 were evaluated. With this aim, the N and P availability was determined on 8 digestates and 2 types of  
17 digestate-based compost from different OW via sequential chemical extractions (SCE). In addition, the  
18 digestates were tested in soil incubations and in plant pot tests with Italian ryegrass and compared with  
19 chemical fertilizer and a non-amended control soil. The N extracted from digestates via SCE was  
20 related to soil N mineralization and plant N recovery. The C: N ratio had negative impact on  
21 mineralized N and its recovery in shoots ( $\text{Shoots}_N = -0.0085 \times \frac{C}{N} + 0.1782$ ,  $r^2=0.67$ ), whereas water  
22 extractable mineral N was positively related to the root N apparent recovery fraction (N-ARF) with  
23 ( $\text{Roots}_N = 5E^{-5} \times N_{\text{solublemin}} + 0.0138$ ,  $r^2=0.53$ ). The shoot P-ARF was positively correlated with the  
24 inorganic water extractable fraction of P ( $\text{Shoots}_P = 0.1153 \times H_2O - P_i - 0.2777 \times H_2O - P_o +$   
25  $0.0249$ ,  $r^2=0.71$ ) whereas the root P-ARF was positively correlated with the less accessible fractions

26  $(\text{Roots}_p = 0.0955 \times \text{NaHCO}_3\text{P}_0 + 0.0955 \times \text{NaOH P}_0 - 0.0584 \times \text{NaHCO}_3\text{P}_1 + 0.0128, r^2=0.8641)$ .

27 Feedstock digestate typology impacted the N and P recovery results leading to a better description of  
28 the typology properties and a first nutrients ARF prediction.

29 **Key words** digestates, typology, fertilizers, characterization indicators, nutrient recovery

30

## 31 **1. Introduction**

32 An increasing interest exists in improving the soil quality following the utilization of organic wastes  
33 (OW) within the environmentally friendly bio-refinery approach (Albuquerque et al., 2012; Gissén et  
34 al., 2014). In this respect, anaerobic digestion is a major building block in the circular bioeconomy.  
35 Furthermore, both energy (ie. biomethane) and organic fertilizers (ie. digestate) replacing the chemical  
36 fertilizers provided to crops can be recovered from anaerobic digestion of OW. From an agronomic  
37 and economic point of view, digestate use as a fertilizer can be considered not only as a supplement  
38 for traditional organic fertilizers (i.e. slurry) but also as an alternative fertilizer, which under certain  
39 soil conditions, is more effective than using NPK fertilizer (Barlog et al., 2019). Indeed, anaerobic  
40 digestion is reported as a suitable treatment to easily recover P and N fertilizers from digestate  
41 (Mazzini et al., 2020). This is due to organic matter and free organic nutrients biodegraded into  
42 mineral forms. However, agricultural reuse of treated OW as soil fertilizers is limited by  
43 environmental constraints related to the quality of organic matter, nutrient availability and safety  
44 issues. Furthermore, the chemical composition of digestate depends on the nature of feedstock and  
45 digestion process (Albuquerque et al., 2012, Guilayn et al., 2019, Barlog et al., 2019). Indeed, a  
46 statistical classification of 91 digestates based on their agronomic quality (i.e. C, N, P, total solids,  
47 organic matter) was applied on a large range of raw digestates by (Guilayn et al., 2019) and revealed 6  
48 groups of different digestate typology. By analyzing the obtained digestates groups, two criteria  
49 impacted the classification: the TS concentration of the digestates associated with dry or wet anaerobic  
50 digestion process and the feedstock type, as following: liquid fibrous feedstock (crop residues silage,  
51 cattle slurry), liquid sewage sludge, liquid pig slurry, slurries co-digested with silage and green wastes

52 (dry anaerobic digestion), municipal solid wastes (dry anaerobic digestion) and fibrous feedstock (dry  
53 anaerobic digestion of cattle manure and green waste). In this framework, mineral N can be readily  
54 available for crops but can be temporarily immobilized by microbial biomass during OW  
55 mineralization in soil (Lashermes et al., 2010). Therefore, it is important to know the OW  
56 mineralization N kinetics to optimize the N supply synchronisation with plant requirement in  
57 agricultural systems.

58 In this context, to better control and manage the quality of digestates, a better knowledge of the  
59 accessibility and availability of nutrients and organic matter would improve the fertilizing potential of  
60 these products. According to Möller et al. (2012), an accurate characterization of digestate nutrient  
61 content and organic matter (OM) composition, combined with experiments to assess the N  
62 mineralization and N immobilization processes after field spreading, would be essential for a better  
63 characterization of the driving factors driving N turnover in the soil.

64 Total content of nutrients in OW hardly predicts their fertilizer potential. Ahmad et al. (2018) reported  
65 more than 80% of applied P is rapidly immobilized being unavailable for plant following  
66 adsorption/precipitation processes or conversion into organic form. To gain a better insight on this  
67 issue, many authors proposed to characterize the available P via P fractionation. He et al. (2010)  
68 reported that bioavailability of applied P depends on the presence of specific P forms and that labile P  
69 includes the sum of inorganic and organic P from H<sub>2</sub>O and NaHCO<sub>3</sub> extracted fractions. In this regard,  
70 Grigatti et al. (2015; 2017; 2019), performed a dedicated SCE on digestates and composts in order to  
71 measure the phosphorous potentially available for plants. The authors found good correlations  
72 between the water- and the sodium bicarbonate-extractable P from composts with short- and medium-  
73 term plant P uptake.

74 Lashermes et al. (2010) proposed an OW classification based on their chemical characteristics to  
75 predict N availability. However, only 2% of the 273 OW studied came from similar typology of  
76 digestates coming from fibrous feedstock anaerobic digestion with low N availability (i.e. digestates  
77 was classified in a group with initial N concentration under 65 g kg<sup>-1</sup> and C: N ratio around 15).  
78 However, according to Guilayn et al. (2019), the N content from digestates varies from 20 g kg<sup>-1</sup> to  
79 175 g kg<sup>-1</sup> and C: N ratio varies from 2 to more than 30. It would be interesting to complete the

80 Lashermes study with different typologies of digestate to classify their fertilizer potential. To do that,  
81 the first step would be to evaluate the impact of digestate typology on plant growth. Recently, a  
82 methodology proposed for organic matter characterization has been applied to a large range of OW in  
83 order to predict both bio-availability and complexity/biodegradability of the organic matter (Jimenez et  
84 al., 2015). This technique has been successfully used for OM biodegradation kinetics modelling  
85 (anaerobic digestion, compost, soil) (Jimenez et al., 2017) and for organic N dynamics in anaerobic  
86 digestion (Bareha et al., 2018; 2019). The basic idea was to find a consistent characterization method  
87 in order to describe the bioprocess kinetics and to model the whole treatment chain in terms of organic  
88 carbon fate, e.g. anaerobic digestion, compost and organic matter fate in soil. The methodology used is  
89 based on SCE to simulate organic matter bioaccessibility for microorganisms combined with three-  
90 dimensional fluorescence spectroscopy to analyze the organic matter complexity. The challenge is  
91 now to transfer this technique to the characterization of nitrogen accessibility in order to predict its  
92 fate after land-spreading and its availability for plant growth.

93 Apart from the previously mentioned studies, there is no literature information on the development of  
94 indicators for both N and P plant availability from digestates. This is the reason this study aimed to  
95 propose indicators based on chemical extractions and characterization for N and P.

96 In this light, the objectives of this work were to determine the plant-available N and P fractions based  
97 on characterization of the accessibility study in order to: (i) assess the impact of typology of OW on  
98 the nutrient availability, and (ii) use these indicators to predict plant's nutrients recovery. This study,  
99 focussed on a wide range of anaerobic digestates from many types of OW. The results could be used  
100 for rapid diagnostic of the (N, P) fertilization potential of a digestate.

## 101 **2. Materials and methods**

### 102 **2.1. Organic waste**

103 In order to ensure a representative survey, the anaerobic digestates used in this work were selected on  
104 the basis of the results reported by Guilayn et al. (2019). The typology developed by (Guilayn et al.,  
105 2019) was used to select the digestate samples out of a large panel of digestates. For this study, 6 main

106 groups were selected from a statistical study applied to the agronomic characteristics: sewage sludge,  
107 municipal waste, cow manure (dry -anaerobic digestion), pig manure (liquid anaerobic digestion),  
108 centralised (co-digestion), and crop residues. Table 1 presents the samples used with the type of OW  
109 used as feedstock and the conditions of the anaerobic digestion process. Post-treated digestates  
110 through phase separation (solid or liquid) and composting were also added. Indeed, phase separation is  
111 the most classical digestate post-treatment (Alburquerque et al., 2012), leading to two products of  
112 different quality (i.e. solid phase as soil amendment and liquid phase as fertilizer). Besides, two  
113 digestate-based types of compost (FFMSW\_2 and Sludge\_2) were used since composting is widely  
114 adopted treatment for the solid phase of urban OW digestate to meet the European fertilizing  
115 regulation parameters (European Parliament and Council of the European Union, 2016). The organic  
116 waste samples were freeze-dried and ground ( $\phi$  1mm), in order to reduce the particle size effect in the  
117 soil incubations and plant growth experiments.

## 118 **2.2. Analytical measurements**

119 Freeze-dried ground samples were further ball milled and analysed for the main physico-chemical  
120 parameters. The moisture was determined at  $105 \pm 2^\circ\text{C}$  until constant weight (24-48h), the volatile  
121 solid (VS) was determined on the total solids (TS) at  $550^\circ\text{C}$  for 4 h. The total carbon (TC) and total  
122 nitrogen (TN) were determined via an elemental analyser (FlashSmart, Thermo Fisher Scientific). The  
123 total nutrient and trace elements were determined by ICP (Inductively Coupled Plasma-OES, Spectro  
124 Arcos, Ametek) on  $\approx 250$  mg of samples after microwave assisted digestion with 65%  $\text{HNO}_3$  + 37%  
125 HCl. All the analyses were done in duplicates.

126 Based on Jimenez et al. (2015), the sequential chemical extractions of organic matter were applied on  
127 the freeze-dried and grounded samples (0.5 g). An orbital shaker was used for the extractions steps as  
128 following described: 30 mL of 0.01M  $\text{CaCl}_2$  (twice, 1h,  $30^\circ\text{C}$ , 300 rpm) ; 0.01M (NaOH+NaCl) (4  
129 times, 15 min,  $30^\circ\text{C}$ , 300 rpm); 0.1M HCl (once, 1h,  $30^\circ\text{C}$ , 300 rpm), 0.1M NaOH (4 times, 1h,  $30^\circ\text{C}$ ,  
130  $300$  rpm) and 72%  $\text{H}_2\text{SO}_4$  (twice, 3h,  $30^\circ\text{C}$ , 300 rpm). The fractions names are respectively:  
131 Soluble extracted from Particulate Organic Matter (SPOM), Readily Extractible Organic Matter  
132 (REOM), Slowly Extractible Organic Matter (SEOM) and Poorly Extractible Organic Matter

133 (PEOM). The not extracted fraction is called Non Extractible Organic Matter (NEOM). Each  
 134 extraction was done in two replicates, centrifuged and the supernatant collected and filtered. The  
 135 recovered pellets were submitted to the subsequent extraction. Ammonium and total nitrogen were  
 136 measured in the supernatants using HachLange® kits based on colorimetry methods (LCK 303, 2-47  
 137 mg N L<sup>-1</sup>, and LCK 238, 5-40 mg L<sup>-1</sup> respectively).  
 138 The organic products were submitted to P fractionation via SCE according to the method of Dou et al.  
 139 (2000). Freeze-dried and ball-milled products were extracted for 24 h with deionized water (H<sub>2</sub>O) in  
 140 an end-over-end shaker, and then centrifuged. The supernatants were passed through a Whatman #42  
 141 filter, and the recovered pellets were extracted with 0.5 M NaHCO<sub>3</sub> (pH 8.5) for 24 h. The same  
 142 procedure was repeated with 0.1N NaOH and 1N HCl. The residual pellets were treated with a mixture  
 143 of 96% H<sub>2</sub>SO<sub>4</sub> and 30% H<sub>2</sub>O<sub>2</sub> via hot acid digestion at 360 °C. Inorganic P (P<sub>i</sub>) in the extracts was  
 144 determined via the molybdenum blue method of Murphy and Riley (1962). The P recovered in each  
 145 fraction [water extractable P (H<sub>2</sub>O-P), bicarbonate extractable P (NaHCO<sub>3</sub>-P), alkali extractable P  
 146 (NaOH-P), acid extractable P (HCl-P), Residual-P] was calculated as follows:

$$147 \quad P_{i \text{ fraction } x} (\%) = \frac{P_{i \text{ fraction } x}}{P_{\text{tot OW}}} \times 100 \quad \text{Equation 1}$$

148 where  $P_{i \text{ fraction } x}$  is the inorganic P determined in each fraction (H<sub>2</sub>O, NaHCO<sub>3</sub>, NaOH, HCl, Residual),  
 149 and  $P_{\text{tot OW}}$  is the total-P determined in the different organic waste via ICP after microwave-assisted  
 150 acid digestion.

151 The total recovery was calculated as the sum of all fractions (H<sub>2</sub>O-P + NaHCO<sub>3</sub>-P + NaOH-P + HCl-P  
 152 + Residual-P) by using the Equation2:

$$153 \quad \text{Tot } P_{\text{recovery}} (\%) = \frac{\sum_{\text{H2O}}^{\text{Residual}} P_{i \text{ fraction } x}}{P_{\text{tot OW}}} \times 100 \quad \text{Equation 2}$$

154 where  $\sum_{\text{H2O}}^{\text{Residual}} P_{i \text{ fraction } x}$  represents the sum of the single P recovery values (P<sub>i</sub>) in each fraction (H<sub>2</sub>O-P +  
 155 NaHCO<sub>3</sub>-P + NaOH-P + HCl-P + Residual-P), and  $P_{\text{tot OW}}$  is the total P determined in the different  
 156 products via ICP after microwave-assisted acid digestion. Organic P was calculated as the difference  
 157 between  $P_{\text{tot}}$  and  $P_i$  in the first four fractions.

158 According to He et al. (2010), H<sub>2</sub>O-P, NaHCO<sub>3</sub>-P, NaOH-P and HCl-P are respectively water-soluble,  
159 bioavailable, potential bioavailable (Fe/Al bound) and Ca-bound P.

160

### 161 **2.3. X-ray diffraction**

162 Samples submitted to X-ray diffraction analysis were carefully ground and placed in a flat stage  
163 sample holder. Data were collected by means of a PANalytical X'Pert PRO powder diffractometer  
164 equipped with a fast X'Celerator detector. Cu K $\alpha$  radiation was used (40 mA, 40 kV). The 2 $\theta$  range  
165 was from 5° to 60° with a step size of 0.1° and time/step of 100 s. Data were processed for phase  
166 identification using a HighScore Plus software package (PANalytical).

167

### 168 **2.4. Soil incubation**

#### 169 **2.4.1. Soil**

170 The soil used for the incubation and pot trial was collected from the top layer in a field in the Po  
171 Valley (Bologna, Italy). This soil showed the following characteristics: pH (H<sub>2</sub>O 1:2.5), 7.90; particle-  
172 size distribution; 184 mg kg<sup>-1</sup>, sand, 425 mg kg<sup>-1</sup>, silt, 391 mg kg<sup>-1</sup> clay; total CaCO<sub>3</sub>, 85 g kg<sup>-1</sup>; total  
173 organic carbon (TOC), 10.2 g kg<sup>-1</sup> ; total Kjeldahl nitrogen (TKN), 1.60 g kg<sup>-1</sup>; C:N, 8.3; exchangeable  
174 K, 330 mg kg<sup>-1</sup> as K<sub>2</sub>O, CEC 27.2 meq. 100 g<sup>-1</sup>. The total (extractable in aqua regia + HF) Al, Fe and P  
175 were 35661, 22224 and 808 mg kg<sup>-1</sup> respectively. The NH<sub>4</sub>-oxalate (pH 3) extractable Al and Fe were  
176 764 and 2158 mg kg<sup>-1</sup> respectively, while the Na dithionite-citrate extractable Al and Fe were 281 and  
177 2462 mg kg<sup>-1</sup> respectively.

#### 178 **2.4.2. Incubation tests**

179 Soil incubation was conducted in 500 ml plastic vessels with perforated plastic cap, on 250 g of soil  
180 (TS basis). The soil was rewetted at 60 % of the water holding capacity (WHC) and pre-incubated for  
181 4 weeks at 25 °C in the dark. Water content was kept at 60% of WHC by weighing the vessels every  
182 2-3 days and by adjusting with water drops if needed. After this period, the organic products (i.e.  
183 digestates) were added to the soil at 170 kg N ha<sup>-1</sup>, defined as the maximum N load per year

184 authorized by the European Nitrates Directive (1991) and accurately mixed by hand. Chemical  
 185 references used as positive control (Ctrl<sup>+</sup>) were added by distributing N (as NH<sub>4</sub>NO<sub>3</sub>) at the same  
 186 concentration of 170 kg N ha<sup>-1</sup>, and in addition P and K were added at 117 and 147 kg ha<sup>-1</sup> (as  
 187 KH<sub>2</sub>PO<sub>4</sub>). On the basis of N loading the P added to the soil with the different organic product was on  
 188 average 20.45 +/- 2.5 mg P kg soil<sup>-1</sup>. Calculation of the amount of digestate mass added is described  
 189 by the Equations 3 to 5 considering 0.3 m of soil depth (d) and a bulk density of 1.33 g/cm<sup>3</sup> for 1 ha of  
 190 soil.

$$191 \text{ dosis} \left( \frac{\text{mgN}}{\text{kg soil}} \right) = \frac{\text{dosis} \left( \frac{\text{mgN}}{\text{ha}} \right)}{\text{area}_{1 \text{ ha}} \times d} = \frac{170 \times 10^6}{10^4 \times 0.3 \times 1.33 \times 10^3} = 44 \text{ mgN} \cdot \text{kg}^{-1} \quad \text{Equation 3}$$

$$192 \text{ dosis} \left( \frac{\text{gN}}{\text{kg soil}} \right) = \frac{m_{\text{digestate}} \times N_{\text{digestate}} \times \text{TS}_{\text{digestate}}}{m_{\text{soil}}} \quad \text{Equation 4}$$

193 From the Equation 4, digestate amount is calculated as:

$$194 m_{\text{digestate}} = \frac{\text{dosis} \times m_{\text{soil}}}{N_{\text{digestate}} \times \text{TS}_{\text{digestate}}} \quad \text{Equation 5}$$

195  $m_{\text{digestate}}$  is the digestate mass (kg)

196  $N_{\text{digestate}}$  is the N concentration of the digestate (gN.kgTS<sup>-1</sup>)

197  $\text{TS}_{\text{digestate}}$  is the Total solids content of the digestate (% of fresh matter)

198  $m_{\text{soil}}$  is the considered soil mass in the test (kg)

199 Three replicates for each treatment were assessed in a completely randomized block design, in  
 200 addition to an unfertilized treatment control (Ctrl<sup>-</sup>). Two parallel soil sampling series were done to  
 201 follow the P and N evolution during incubation. Olsen-P, was determined according to Watanabe and  
 202 Olsen (1965) at days: 0, 14, 28, 56, 84. The Olsen-P data were used to calculate the relative percentage  
 203 extractable (RPE) P, used to normalise the P extractability for each treatment relative to Olsen-P  
 204 obtained for Ctrl<sup>+</sup> (Grigatti et al., 2019). The mineral N course in soil was assessed by extracting soil  
 205 samples (1g TS basis) at days 0, 14, 28, 56 and 84 with 1M KCl for 30 min on an end-over-and end  
 206 shaker. The solution was filtered over a Whatman #42 filter and ammonium and nitrates were

207 determined using Hach Lange® kits (LCK 304, 0.015-2 mg N.L<sup>-1</sup> and LCK 339, 0.23-13.5 mg N L<sup>-1</sup>  
208 respectively).

## 209 **2.5. Plant pot trials**

210 Based on Grigatti et al. (2014 ; 2015), the digestate samples were added to the ground soil at 170 mg  
211 N kg<sup>-1</sup> and thoroughly mixed by hand in 2 liter plastic pots (ø 140 mm × h 150 mm). These were filled  
212 with 1 liter of inert material (agricultural light expanded clay), and 1 kg of each different treated soil in  
213 3 replicates. Pots were seeded with 0.8 g of seeds of Italian ryegrass (*Lolium multiflorum* subsp.  
214 *Italicum*), cv. Sprint, covered with a thin layer of sand to prevent drying, watered and placed in a  
215 growth chamber at 16-18 h day-night photoperiod at 13–23 °C (±3 °C) day-night temperature, the light  
216 was ensured by 6 Philips Master Tld 58 W-840 tubes. Besides the organic products, an unfertilized  
217 control (Ctrl<sup>-</sup>), and a chemical reference (at the same rate used for soil incubation) was added (Ctrl<sup>+</sup>).  
218 The same treatments and dosis performed in soil incubation test were applied. The use of fast growing  
219 species as ryegrass in controlled conditions (moisture; temperature; light) leads to a multiple harvest  
220 approach giving the opportunity to the best description of apparent N and P utilization kinetics in the  
221 time frame of a growing season (Gunnarson et al., 2010; Schiemenz et al., 2010 ; Tampio et al., 2016).  
222 After emergence, plants were regularly watered with tap water to keep soil at 60 % Water Holding  
223 Capacity (WHC). At each harvest, ryegrass plants were cut 2 cm above ground and collected 3 times:  
224 at 28, 56 and 84 days after sowing. The plant biomass was then dried in a forced air oven at 60 °C for  
225 3 days and weighed, to determine dry weight (DW) per pot. Dry biomass was also ball-milled for  
226 subsequent analysis. At the last harvest, the roots were divided from the soil with a combined water-  
227 sieving separation, and the root biomass was then dried as above, weighed and milled. On plant tissue  
228 (shoots and roots), the total P content was determined by means of ICP after (HNO<sub>3</sub>, H<sub>2</sub>O<sub>2</sub>,)  
229 microwave assisted digestion. The total N content was determined by elementar analysis as the OW  
230 samples. Apparent plant N and P utilization efficiency was calculated on the basis of the Apparent  
231 Recovery Fraction (ARF) approach (Gunnarson et al., 2010), according to the Equation 6:

$$232 \text{ ARF}_X (\%) = \frac{\sum_{i=1}^3 \text{X uptake treatment}_{ti} - i \text{X uptake ctrl}^{-}_{ti}}{\text{X added}} \quad \text{Equation 6}$$

233 in which X uptake treatment ( $t_n$ ) is the total nitrogen or phosphorus uptake ( $\text{mg pot}^{-1}$ ) of a fertilizer  
234 treatment at time t ( $i = \text{cut 1-3.}$ ); X uptake ctrl<sub>i</sub> is the total nitrogen or phosphorus uptake ( $\text{mg pot}^{-1}$ ) of  
235 the unfertilized control at time t ( $i = \text{cut 1-3.}$ ); X added is the total nitrogen or phosphorus added to the  
236 pot ( $\text{mg pot}^{-1}$ ).

237 Chemical nutrient equivalent coefficient  $k_{eq}$  is defined as the equivalent chemical nutrient dosis (i.e.  
238 positive control) percentage needed to reach similar crop yield and is calculated according the  
239 Equation 7.

$$240 \quad k_{eq}X = \frac{ARF_X}{ARF_{Ctrl+}} \quad \text{Equation 7}$$

## 241 **2.6. Statistical analysis**

242 All the data from the plant growth experiment were analyzed by means of Kruskal-Wallis non  
243 parametric test. Principal Component Analysis (PCA) and Hierarchical Clustering Analysis (HCA)  
244 were also performed on all the data using FactomineR package from the R software. Partial Least  
245 Square Regression (PLSR) was performed for variables prediction using the SIMCA® software. For  
246 PCA analysis, data from Grigatti et al. (2019) were included as far as similar experiments were done  
247 on P speciation and plant pot tests. The three samples were D1, D2 and BD, which are respectively  
248 digestate from thermophilic wastewater sludge digestion, digestate from mesophilic winery sludge  
249 treatment and digestate from mesophilic bovine slurry and energy crops treatment. Table 1 presents  
250 these samples, the conditions of the anaerobic digestion process and the associated feedstocks.

## 251 **3. Results and discussion**

### 252 **3.1. Digestates characterization**

253 The main physico-chemical characteristics of the studied digestates are reported in the Table 2. A high  
254 variability of the parameters analysed was apparent. Indeed, organic matter ranged between 40 and  
255 and 90% (TS basis), the TKN varied between 15 and 45  $\text{g kg}^{-1}$  while P ranged between 4 and 20  $\text{g kg}^{-1}$ .  
256 The most N-rich samples (44-48  $\text{g kg}^{-1}$ ) were the liquid phase of the centralised digestate (Centr\_2),  
257 the pig manure digestate (Agri\_2) and the sludge digestates (Sludge\_1, D1 and D2). The poorest

258 samples were the digestates of FFMSW and its compost (between 15 and 17 g N kg<sup>-1</sup>). The richest P  
259 samples were the sludge digestate (Sludge\_1) and its compost (Sludge\_2), along with the solid phase  
260 of the centralised digestate (Centr\_1) and digestate D2 (sludge from winery processing) with a P  
261 content ranging between 18 and 20 g kg<sup>-1</sup>. The FFMSW\_1 digestate and its compost FFMSW\_2  
262 besides the silage straw digestate (Agri\_3) were the poorest P samples (4 g P kg<sup>-1</sup>). PCA and HCA  
263 analyses were performed on the samples characteristics for a whole sight of the digestates profiles thus  
264 allowing the comparison with the typology of Guilayn et al. (2019). The results are presented in the  
265 Supplementary Material (Figure A.1). The PCA was applied on nine parameters (i.e from TS to K in  
266 Table 2). The first two components recovered  $\approx 80\%$  variance. The first component (PC1) was mainly  
267 related to the TS, negatively related to OM and K.

268 PC1 clustered dried (i.e. FFMSW\_1, composts Sludge\_2 and FFMSW\_2) and liquid AD digestates  
269 (i.e. all the others). The second component (PC2) was formed by P, which was negatively related to  
270 the C: N ratio related to fibrous composition. The sludge digestate samples were associated with P  
271 concentration variable whereas Agri\_3 (wheat straw digestate) was mainly associated to C: N ratio  
272 variable. Furthermore, Sludge\_1 and 2 were the samples containing the highest metals concentrations  
273 (Fe, Al, Pb, Cu, Mn, Mo, and Cd).

274 These results were in agreement with the raw digestate typology found by Guilayn et al. (2019).  
275 Indeed, Agri\_1 and Agri\_3 characteristics were in agreement with the characteristics associated to the  
276 fibrous feedstock digestate group after dried digestion (i.e. cattle manure and crop residues). Agri\_2  
277 characteristics were consistent with those of the pig slurry digestate group from the typology.

278 Sludge\_1 characteristics were consistent with those of the sludge digestate group and FFMSW\_1  
279 characteristics fit with the municipal solid wastes digestate group characteristics. BW\_1  
280 characteristics were mainly close to the biowaste and municipal solids waste digestate composition  
281 after dry digestion. However, NH<sub>4</sub> concentration and NH<sub>4</sub>: TN ratio had similar values than the  
282 manure co-digestion (i.e. NH<sub>4</sub>: TN ratio > 70%). This is probably due to the high ratio of agro-  
283 industrial waste in the feedstock used in addition of biowastes. Only digestate from the wet digestion  
284 of fibrous substrate was not taken into account (silage) in this study. Composts Sludge\_2 and

285 FFMSW\_2 and Centr\_1 and 2 obtained after phase separation were not compared with the typology as  
286 they were not raw digestates.

287 Attention should be paid to the results for TKN and N-NH<sup>+</sup><sub>4</sub> concentration obtained by the raw sample  
288 analysis and to the freeze-dried sample extraction analysis. Regarding some digestates, highly  
289 significant positive differences were obtained between raw and freeze-dried samples analyses (from  
290 40% for Centr\_2 to 61% for Agri\_2 of TKN loss). This result was due to ammonia volatilization  
291 during freeze-drying operation. The loss of ammonia could have an impact on soil incubation and  
292 plant N recovery results. Considering the 8 others digestates, no significant differences appeared and a  
293 linear relation was obtained between raw sample analysis and freeze-dried analysis (i.e.  $TKN_{raw} =$   
294  $0.9443 \cdot TKN_{freeze-dried}$ ,  $R^2 = 0.8832$ ). Furthermore, the N typology of Agri\_2 became similar as  
295 Agri\_1 and 3 (fibrous feedstock involving manures). NH<sub>4</sub>: TN ratio evolved from 76% to 39%. This  
296 ratio decreased also for Centr\_2 from 46% to 9% after freeze-drying. Consequently, Agri\_2 and  
297 Centr\_2 results were used in statistical tests by using the freeze-dried analysis. N-ARF recovery will  
298 be discussed accordingly. However, concerning liquid digestates with high ammonia content as pig  
299 slurry digestates, the best option would be to perform soil incubation and plant pot trials with fresh  
300 matter as Rigsby et al. (2013), de la Fuente et al. (2013). A higher mass of soil would be required to  
301 maintain the soil WHC with the liquid digestate moisture, according Equation 5.

302 This first characterization approach showed that the digestates selected were representative of a large  
303 range of digestates (i.e. municipal waste, sludge, manure, crop residue, bio-wastes, centralised).

### 304 **3.1.1. X-ray powder diffraction**

305 The X-ray powder diffraction has been widely use for the study of the crystalline P species in anaerobic  
306 digestate and compost (Li et al., 2019; Grigatti et al., 2017; 2019). In this light the XRD profiles from the  
307 tested products can give a valuable insight to their P extractability. The XRD patterns from the digestates  
308 are presented in Figure 1. The XRD patterns of samples Agri\_1, Agri\_2 and Agri\_3 were similar  
309 showing the presence of crystalline inorganic phases, namely quartz (PDF no. 01-087-2096), calcite  
310 (PDF no. 01-085-1108) and sylvine (KCl, PDF no. PDF no.41-1476).

311 However, these three samples differed significantly in their amorphous phase content, which was  
312 definitively higher in Agri\_3, followed by Agri\_1, whereas sample Agri\_2 was mostly constituted of  
313 crystalline material. Furthermore, Agri\_2 presents also struvite peaks (PDF no. 15-0762), that could be  
314 detected on account of the higher crystallinity of this sample. Amongst the analyzed samples, Sludge\_1  
315 was the most crystalline one, with no significant presence of amorphous material. The Sludge\_1 pattern  
316 showed the presence of calcite and quartz as main constituting phases, together with a low amount of  
317 vivianite ( $\text{Fe}_3(\text{PO}_4)_2(\text{H}_2\text{O})_8$ , PDF no. 01-075-1186). Li et al. (2018) found also that quartz was one of the  
318 major phases for sewage sludge. Sludge\_2 was somewhat similar to Sludge\_1, but less crystalline so that  
319 only calcite and quartz were detected (in this case quartz is more abundant than calcite). FFMSW\_1 and  
320 FFMSW\_2 were very similar both regarding the amount of amorphous phase which is present together  
321 with the fine crystalline fraction of the materials. Regarding the qualitative detection of crystalline  
322 phases, it was possible to identify quartz (the most abundant), calcite, together with low amounts of  
323 kaolinite ( $\text{Al}_2(\text{Si}_2\text{O}_5)(\text{OH})_4$ , PDF no.01-080-0885) and sylvite. BW\_1, Centr\_1 and Centr\_2 patterns all  
324 displayed the presence of quartz, calcite and struvite as crystalline components, but significantly differed  
325 for the amount of amorphous material that was relatively low in Centr\_2, and higher and similar in  
326 BW\_1 and Centr\_1. The relationship between the XRD outcomes and the P extractability are discussed  
327 in the following section.

### 328 **3.1.2. Sequential chemical extractions of Nitrogen and Phosphorous**

329 The N fractionation protocol used in this work was developed by Jimenez et al. (2015), and has been  
330 validated to describe OM and N evolution during biological degradation processes (Jimenez et al.,  
331 2017 and Bareha et al., 2018). The P fractionation has been adapted from Hedley et al. (1982), using  
332 methodology by Grigatti et al. (2015, 2017, and 2019). These authors showed the link between labile-  
333 P from organic waste samples and the plant P-uptake. The N and P speciations have been assessed  
334 applying both fractionation protocols to the selected digestates (Figure 2 a and b respectively). The  
335 results showed that the digestates performed different N and P speciations. This was related to the  
336 digestate typology and the nature of the feedstock for anaerobic digestion, according to Guilayn et al.

337 (2019). The observed variability in N and P speciation showed the significant effect of the feedstock on  
338 the tests discussed in section 3.2. In the study of N accessibility (Figure 2 a), mineral nitrogen was  
339 assessed in the water extractable fraction SPOM. The non-extractable organic nitrogen varied between  
340 25 and 48% except for Agri\_3 with only 5% N in the NEOM fraction.

341 HCA was applied on the N speciation data (not shown). Four groups were found, ordered by N  
342 accessibility basis as follows: (i) Group 1: High NH<sub>4</sub>-N SPOM samples: Centr\_1 and BW\_1. This  
343 result is consistent since these samples had poor TS; (ii) Group 2: High organic SPOM and REOM:  
344 Agri\_2 and Sludge\_1. SPOM and REOM are mainly composed of accessible and readily extractable  
345 proteins; (iii) Group 3: High organic SEOM: Agri\_3. SEOM is mainly composed of complex proteins  
346 and humic-like substances; (iv) Group 4: High PEOM organic N content samples: Agri\_1, FFMSW\_1,  
347 Centr\_2, FFMSW\_2 and Sludge\_2. PEOM extraction targets holocellulose-like compounds found in  
348 fibrous digestates as Agri\_1, FFMSW\_1 and 2. Phase separation concentrates OM and fibers in the  
349 solid phase (Guilayn et al., 2019) as Centr\_2. Finally, the compost Sludge\_2 was also clustered in this  
350 group because of its co-composting with green wastes.

351 P was extracted in each fraction as reported in Figure 2 b. Phosphorus fractionation showed that  
352 inorganic P content was higher than organic P in all the fractions, as reported in the studies of Grigatti et  
353 al. (2015, 2017 and 2019) on digestates and composts, and as observed by He et al. (2010) on poultry  
354 litter and dried wastewater sludge. Mazzini et al. (2020) showed that 78 to 93% of TP was inorganic  
355 following the SCE from six types of animal slurries digestates. The authors showed relevant NaOH (5-  
356 43% of organic P) and HCl (2-25% of organic P) extractable organic P, relating this to inorganic P  
357 microbial immobilization during anaerobic digestion for microorganism growth. Indeed, soluble  
358 inorganic P would be transformed into organic P compounds such as phosphates monoesters or DNA.  
359 This biological organic-P may be rapidly mineralized once in soil (He et al., 2010). In this context,  
360 NaOH and H<sub>2</sub>O were the most organic-P rich fractions from the samples investigated in this work.  
361 Furthermore, Agri\_1, 2 and 3, and FFMSW\_1 mainly showed NaOH extractable organic P. Agri\_3  
362 contained the highest organic P content ( $\approx$ 40%), and 1.5 to 4- folds higher than other samples. In water  
363 Agri\_2 and 3 showed 60% of organic P, higher than BD (49%).

364 The results obtained by He et al. (2010) on poultry litter showed that a large part of organic P was in the  
365 NaOH fraction and was mainly related to phytate-like. Mazzini et al. (2020) showed similar results on  
366 crop residues digestates. The authors showed also that organic P were mainly extracted in NaOH fraction  
367 of 6 agro-wastes digestates as shown by Agri\_1 and 3. Organic P was observed in HCl fraction from  
368 Sludge\_1 and 2 in this study. He et al. (2010), observed also organic P in HCl fractions related to non-  
369 hydrolysable fractions in the dried wastewater sludge. The NaHCO<sub>3</sub> and the HCl fractions showed to be  
370 the most inorganic P rich fractions from the samples tested in this work thus fitting to available P and Ca-  
371 bounded P. The labile-P fraction (H<sub>2</sub>O + NaHCO<sub>3</sub>) was found at the highest level in the agricultural  
372 residue digestates (Agri\_1, 2 and 3). Similar observations on bovine manure and energy crops digestate  
373 (BD) were made by Grigatti et al. (2019). Then the biowaste and agro-food industries digestates (BW\_1,  
374 Centr\_1 and 2) had also high labile-P. The FFMSW samples showed intermediate P accessibility. The  
375 sludge samples were characterized by a high NaOH extractable P, related to metal-bounded P. Indeed,  
376 this result was consistent with both Al and Fe content ( $\approx 7$  and  $\approx 37$  g kg<sup>-1</sup>). Li et al. (2018), showed  
377 mainly NaOH extractable Al, while Fe was NaOH soluble being also extracted by HCl in sewage sludge,  
378 thus showing Fe-bounded P was partially occluded. Amongst the others samples, compost Sludge\_2  
379 showed important poorly available HCl-P, being very similar to FFMSW\_2, these results were consistent  
380 with the P fractionation of composts showed by Grigatti et al. (2015, 2017, and 2019).

381 Finally, HCA showed six groups (clustering not shown). Groups were ordered according to the chemical  
382 availability level (i.e. Labile-P > NaOH-P > HCl-P) as follow: (i) Very high P accessibility related to high  
383 H<sub>2</sub>O-P fractions: digestates from liquid feedstock (no manure), biowastes and winery (BW\_1, D1 and  
384 D2). These samples were characterized by amorphous P and crystalline P (quartz and struvite); (ii) High  
385 P accessibility associated with organic H<sub>2</sub>O-P and NaHCO<sub>3</sub>-P: digestates from liquid manure (BD and  
386 Agri\_2). These samples were characterized by crystalline P (struvite); (iii) Moderate P accessibility  
387 (intermediary P speciation): digestates from organic fraction from municipal wastes digestates and  
388 centralised digestates (FFMSW\_1, FFMSW\_2, Centr\_1 and Centr\_2). These samples were characterized  
389 by an amorphous phase and mainly quartz and calcite as crystalline phases; (iv) Poor-P accessibility  
390 associated with organic NaHCO<sub>3</sub>-P and NaOH-P, associated with digestates from fibrous substrates as  
391 cow manure with crops residues and wheat straw (Agri\_1 and Agri\_3). These samples were

392 characterized by a high amorphous phase; (v) Very poor P accessibility associated with inorganic NaOH-  
393 P and low fraction of available P: digestate from solid phase of sludge (Sludge\_1). This sample was  
394 characterized by a crystalline P phase (mainly quartz and calcite); (vi) The poorest accessible P  
395 associated with inorganic HCl-P was compost of sludge digestate (Sludge\_2). This sample was  
396 characterized by less crystalline P than Sludge\_1 with calcite and quartz.  
397 For some groups, the feedstock type seemed to have an impact on the P accessibility of digestates (solid  
398 phase of sludge, liquid manure, liquid feedstocks, fibrous feedstock). Another observation was that the  
399 amorphous and crystalline characteristics also seemed to be associated with some groups: fibrous  
400 digestates were mainly composed of amorphous phase and contained organic P in NaHCO<sub>3</sub> and NaOH.  
401 The labile P fractions had in common amorphous P and struvite as crystalline P. The most crystalline P-  
402 rich fractions were found in the solid phase of sludge digestates before and after composting, containing  
403 low available P and high sparingly soluble HCl-P.  
404 The eight tested digestates represented a wide range of inherent characteristics and accessibility patterns  
405 for both N and P. In this framework, the soil and plant test results can give a deeper insight to these  
406 issues as discussed in the following section.

### 407 **3.2. Soil incubation and plant pot trials**

408 Table 4 shows the cumulative amount of biomass harvested in ryegrass plant pot tests. Any significant  
409 differences between treatments for tissue and root (Kruskal-Wallis test  $p=0.89$  and  $0.23$  respectively).  
410 Nevertheless, it appears that poor biomass was obtained in Agri\_2 treatment (pig manure digestate),  
411 and in FFMSW\_2 treatment (FFMSW digestate compost), close to the negative control. The best  
412 results were obtained in Centr\_1 and BW\_1 (solid phase of a centralised digestate and biowaste  
413 digestate). The total plant biomass (shoot+ root) was the highest ( $\text{g pot}^{-1}$ ) in Centr\_1 ( $4.11$ )  $\geq$  Sludge\_2  
414 ( $3.75$ )  $\geq$  BW\_1 ( $3.72$ )  $\geq$  FFMSW\_1 ( $3.70$ )  $\geq$  Ctrl+ ( $3.65$ )  $\geq$  Sludge\_1 ( $3.48$ )  $\geq$  Agri\_3 ( $3.38$ )  $\geq$  Centr\_2  
415 ( $3.23$ )  $\geq$  Agri\_\_1 ( $3.18$ )  $\geq$  Agri\_2 ( $3.02$ )  $\geq$  FFMSW\_2 ( $2.93$ )  $\geq$  Ctrl- ( $2.90$ ).

#### 416 **3.2.1. Nitrogen fate**

417 The N mineralization data from soil incubation (Figure 3) showed the net cumulated mineral N (i.e.  
418  $\text{NH}_4 + \text{NO}_3$  mass) during soil incubation (Figure 3a) and the net cumulated mineralization rate of  
419 organic N (Figure 3b).

420 Concerning the available mineral N (N-min), BW\_1, Centr\_2, Sludge\_1, Sludge\_2, Agri\_2 and  
421 FFMSW\_1 achieved the highest cumulated values after 84 days of incubation. On the contrary,  
422 Centr\_1, FFMSW\_2, Agri\_3 and Agri\_1 performed poorly cumulated N-min. These latter were more  
423 fibrous samples, with high C: N ratios.

424 The cumulated organic N mineralization rate was calculated (Figure 3b). Two groups of treatments  
425 appeared and were classified in the same order than the cumulated N-min: (i) positive mineralization  
426 rate associated with the Sludge\_1, Centr\_2, BW\_1, Agri\_2, Sludge\_2 and FFMSW\_1 treatments and  
427 (ii) negative mineralization rate associated with Centr\_1, FFMSW\_2, Agri\_1 and Agri\_3. Sludge\_1  
428 showed the highest N mineralization rate (30%) suggesting that this digestate was rich in hydrolysable  
429 proteins. Organic N from the liquid digestates Centr\_2 and Agri\_2 showed respectively 21% and 18%  
430 of mineralized organic N, following Sludge\_1 (30%). This result is consistent with Guilayn et al.  
431 (2019) who reported that the highest organic N came from sludge digestate and pig-slurry digestate  
432 group. Sludge\_2 and FFMSW\_1 performed lower N mineralization rates (11%). Similar results were  
433 observed in liquid or solid digestates by Rigsby et al. (2013). They showed that the solid phase from  
434 municipal solid wastes digestate (C: N ratio of 11) had negative N mineralization rate whatever the  
435 soil composition used (sandy, silty or clay). On the contrary, Rigsby et al. (2013) showed that the  
436 liquid digestates from slurry (C: N ratio of 4), had 16% to 40% N mineralization while food waste  
437 digestate (C: N ratio of 14) reached 30% N mineralization.

438 The negative mineralization rates group was associated to an immobilization of mineral nitrogen by  
439 the soil microorganisms during their growth (De la Fuente et al., 2013). This was related to the  
440 increased microbial activity following the OW addition to the soil. Indeed, nitrogen is the main  
441 limiting nutrient for plant growth, especially for a fast growing, highly demanding species such as  
442 Italian ryegrass (Grigatti et al., 2011). These results are in agreement with Cavalli et al. (2017) which  
443 reported immobilization for high C: N ratio anaerobic digestates. The authors showed the low C: N  
444 ratio (5 to 7) associated to the raw digestate and its liquid phase (cattle slurry/mais silage) induced net

445 N mineralization ( $\approx 30\%$ ). On the contrary, the cellulose- and volatile fatty acids-rich solid phase (C:  
446 N ratio, 20) induced lower net N mineralization (9–16%). Indeed, negative correlations were found  
447 between the N mineralization rate and C: N ratio ( $r = -0.73$ ,  $p = 0.029$ ) as observed by Morvan et al.  
448 (2006) on animal manure wastes.

449 Considering the impact of process treatment of digestates, composting appeared to negatively affect N  
450 mineralization. N mineralization rate from Sludge\_2 (11%) was lower than Sludge\_1 (30%) and  
451 FFMSW\_1 (11%) had highest mineralized values than FFMSW\_2 (-2%). Phase separation impacted  
452 also organic N mineralization as shown by Cavalli et al. (2017). Solid phase (Centr\_1) reached  
453 negative values of mineralization whereas Centr\_2 reached 21% of mineralized N.

454

455 Plant growth experiments were affected by fertilizer treatment for N-ARF (Kruskal-Wallis,  $p = 0.026$ )  
456 only for shoot plant tissues. Cumulated N recovery by plant tissues was plotted in the Figure 4. The  
457 chemical treatment showed the greatest shoot N recovery (70%) with a total of 76% of N-ARF. N  
458 uptake recovery was mainly observed in shoots (64% to 87% of the total recovery). Root N-ARF was  
459 measured between 7% and 36% of the total N-ARF, except for Agri\_1 and Agri\_3 treatments where  
460 negative to zero values were obtained.

461 Sludge\_1 and Sludge\_2 achieved the best N-ARF amongst the digestates treatments (16.5 and 15.1%).  
462 These results were consistent with the Sludge\_1 treatment yielding the highest N-min available for  
463 plant growth. Centr\_2 (13.7%), BW\_1 (13.2%) and Centr\_1 (11.6%) treatments showed intermediate  
464 total N recovery. The FFMSW\_1, FFMSW\_2 treatments followed with 8.2% and 6.4% respectively.  
465 Finally, a last group formed by Agri\_1 and Agri\_3 was observed with low N-ARF of 1.8% and 2.6%  
466 respectively. In soil incubation tests, Agri\_1 and 3 showed N immobilization. Accordingly to these  
467 outcomes, the plant N-ARF was negative or close to 0 and close to the unfertilized soil treatment.

468 Considering the digestate treatments impact, composting (i.e. FFMSW\_2 and Sludge\_2) lowered the  
469 fertilizing N value of its associated digestate (i.e. FFMSW\_1 and Sludge\_1 respectively). Phase  
470 separation impacted also the N fertilizing potential as shown by Tambone et al. (2017). Centr\_2  
471 treatment induced higher N mineralization rates and N-ARF than Centr\_1 treatment. According to N

472 speciation and characterization, the solid phase Centr\_1 contained more fibers than the liquid phase  
473 Centr\_2.

474 PCA and HCA were performed to find correlations between the N availability data, digestate  
475 characteristics, mineralized N percentages and N-ARF from shoots and roots as reported in Figure A.2  
476 a (supplementary material). No significant correlation was observed between the total N content, the  
477 mineralization rate and the N-ARF. However, a strong correlation was obtained between the C: N ratio  
478 and N-ARF from shoots-N ( $r=-0.82$ ,  $p=0.004$ ) as in the mineralization N tests without plants. This was  
479 described by a linear equation (Equation 8) based on the data of this study and validated by data from  
480 digestates and composts studied in Grigatti et al. (2015). Similarly, Decoopman et al. (2017) found a  
481 correlation between C: N ratio of agricultural digestates and plant N-ARF measured in field  
482 experiments on cereals.

$$483 \text{Shoots}_N = -0.0085 \times \frac{C}{N} + 0.1782, r^2 = 0.6718 \quad \text{Equation 8}$$

484 The fiber component of digestates had a negative impact on mineralised N and N recovery by plant  
485 tissues. Similarly, the fiber fraction PEOM of digestates was negatively correlated with cumulated  
486 mineralized N ( $-0.58$ ,  $p=0.08$ ) and shoots-N although to a lesser extent ( $r=-0.58$ ,  $p = 0.44$ ). Moreover,  
487 cumulative mineralized N and shoots-N ARF were positively correlated ( $r=0.72$ ,  $p=0.02$ ) which was  
488 consistent with the availability of N for plant growth. Concerning roots, there was a positive  
489 correlation between roots N-ARF and nitrates content ( $r=0.72$ ,  $p=0.017$ ), SPOM\_NH<sub>4</sub> ( $r= 0.61$ ,  
490  $p=0.06$ ) whereas SPOM\_org and roots\_N ARF were negatively correlated ( $r=-0.59$ ,  $p=0.07$ ). A linear  
491 equation (Equation 9) was found between Roots N-ARF and water extracted mineral N (i.e.  
492  $N_{soluble\_min} = \text{NO}_3^- + \text{SPOM\_NH}_4$ ).

$$493 \text{Roots}_N = 5E^{-5} \times N_{solublemin} + 0.0138, r^2 = 0.5313 \quad \text{Equation 9}$$

494 According to Gunnarsson et al. (2010), plants respond to N availability with a different root/shoot  
495 nutrient allocations and plant growth rate could be influenced by the ammonia/nitrates ratio in soil  
496 solution due to the different N sources. Authors observed an increase in root biomass as a result of the  
497 availability of a high amount of ammonia.

498 HCA revealed four clusters (Supplementary material Figure A.2.a) as follows: (i) Group 1: Agri 3  
499 associated with fibrous digestate with high C: N (>25), high SEOM\_N and PEOM\_N and low shoots-  
500 N ARF. This type of digestate is not suitable as N fertilizer; (ii) Group 2: FFMSW\_2 and Agri\_1  
501  $15 < C/N < 13$ , intermediary protein-like and fibrous substrate digestates with high organic SPOM\_N  
502 and PEOM\_N for Agri\_1 as compost FFMSW\_2. They recovered very low roots N-ARF and  
503 intermediate level of shoots N-ARF; (iii) Group 3: Centr\_1, FFMSW\_1 and BW\_1 municipal solids  
504 and biowastes digestates, with C: N ratios of 15 in average, high ammonium and nitrate levels in the  
505 labile fraction and high levels of total N recovery; (iv) Group 4: The protein-like digestates (Sludge\_1,  
506 Centr\_2, Agri\_2) which have a poor C: N (<10), poor PEOM\_N, high SPOM\_Norg content and the  
507 highest total N recovery. Sludge\_2 was classified in the Group 4 because of its lower C: N ratio (9.7)  
508 and its high N mineralization level.

509 C: N ratio is associated to the fibrous level of an OW and seemed to be discriminant enough to predict  
510 N-ARF and organic N mineralization rate. Digestates with C: N ratios  $\geq 15$  were not suitable as N  
511 fertilisers whereas digestates with lower C: N ratios had a high potential of N fertilizer.

512 Accessibility fractionation of N allowed a consistent digestate classification. Furthermore, interesting  
513 correlation between roots-ARF N and water extractable nitrates and ammonia were found.

### 514 **3.2.2. Phosphorous fate**

515 The Olsen-P course during the soil incubation is shown by Figure 5a. The Olsen-P evolution in soil  
516 depends on several phenomena: (i) the physico-chemical conditions of the soil can trap some weakly  
517 bound P, (ii) the organic part of P can be mineralized and (iii) P can be taken up by soil  
518 microorganisms for their growth.

519 The control soil had the lowest Olsen-P content throughout the incubation (12-14 mg kg soil<sup>-1</sup>),  
520 whereas the compared treatments exhibited different Olsen-P courses in time. All the tested samples  
521 showed an available P depletion during the first two weeks. The chemical control (Ctrl<sup>+</sup>) performed  
522 the fastest depletion. Indeed, the chemical P treatment (phosphate salt) can be rapidly fixed with Ca  
523 components in calcareous soil (Albuquerque et al., 2012). In this context, the presence of organic

524 matter and the different P forms from digestates can have positive interactions with soil, performing  
525 lower fixation and higher efficiency in comparison to chemical P sources (Albuquerque et al., 2012).  
526 In some treatments, an Olsen-P increase appeared between 14 and 28 days (Agri\_2, Centr\_1, BW\_1  
527 and FFMSW\_1) probably due to organic P mineralization before another decrease until 56 days. This  
528 latter decrease led to a lower quantity of available P for plant growth. The RPE obtained was between  
529 50 and 84%. The RPE of the Centr\_2 treatment was the highest with 84% vs. the chemical P  
530 treatment, followed by Sludge\_2 (74%). FFMSW\_1, Agri\_2 and Agri\_3 were the samples where the P  
531 fixation was the highest (RPE of 60% in average). The other digestates were intermediate between the  
532 two groups. FFMSW\_1, Agri\_2 and Agri\_3 contained high fractions of water P.

533 The pot tests showed the treatments affected the plant P tissue (p-value = 0.021). In this context the  
534 control soil performed the worst at the first cut (1.93 mg.pot<sup>-1</sup>), showing very poor performance during  
535 the plant pot test reaching 4.55 mg pot<sup>-1</sup> on average (Figure 5b and 5c). The shoot-P ARF is shown in  
536 Figure 4a and b. P-ARF from Agri\_2 and Agri\_3 was negative from the beginning. This result was  
537 consistent with the soil incubation observations. This can be linked with high fixation of P in soil as  
538 suggested by Ahmad et al. (2018). In the second cut (day 56), the cumulative shoots P-ARF decreased  
539 in all the treatments. Only FFMSW\_1 and Centr\_2 showed opposite outcomes. Then, between the  
540 second and the third cut (day 56; 84), shoot-P ARF further increased. In the end the whole P-ARF (%)  
541 was: Centr\_1 (5.78)≈ FFMSW(5.31)≥BW\_1 (4.35)>Centr\_2(3.78)>Sludge\_1 (2.72)≥Agri\_1  
542 (2.44)≥Sludge\_2 (2)>FFMSW\_2 (1.6)>Agri\_2 (0.6)≈Agri\_3 (0.5%).

543 FFMSW\_2, Agri\_2 and 3 showed lower shoots P-ARF than the chemical treatment (1.81%). That  
544 means they were not suitable for P fertilization on calcareous soil.

545 The root-P content was unaffected by the treatment. However, the root P-ARF (%) was: Agri\_1  
546 (2.46)> Agri\_3 (2.04)> FFMSW\_1 (1.18) = Sludge\_1 (1.17) = Sludge\_2 (1.16)> Centr\_1 (1.04)>  
547 BW\_1 (0.3)> Centr\_2 (0.00)> FFMSW\_2 (-0.08)>Agri\_2 (-0.21). Shoots-P and roots-P recovery  
548 potential were different depending on the treatment, except for Agri\_2 and FFMSW\_2 which had the  
549 lowest ARF P recovery for both.

550 Briat et al. (2020) reported that availability of soil P (and K) for plants highly depends on the N  
551 availability. They reported that P concentrations on shoots were correlated with N concentrations in  
552 shoots. Indeed, a significant correlation was observed in this study between total N-ARF and total P-  
553 ARF ( $r = 0.8152$ ,  $p = 0.0022$ ) with a linear relationship ( $ARF\_N/ARF\_P = 7.7535$ ,  $r^2 = 0.6626$ ) thus  
554 proving N and P recovery by plants were linked.

555 PCA and HCA analysis were performed on both shoots and roots P uptake and P speciation for the 8  
556 digestates studied beside the three digestates from Grigatti et al. (2019), as shown by Figure A.2 b in  
557 Supplementary Material. Six groups were found according HCA analysis. The results showed that the  
558 P-ARF was classified not only according to the digestate's nature but also according to their P  
559 accessibility. The groups found were: (i) group 1 : Sludge\_2, compost of solid phase of sludge  
560 digestate, HCl rich with an intermediate level of P recovered in shoots and roots; (ii) group 2 :  
561 Sludge\_1, solid phase of sludge digestate (inorganic  $NaHCO_3$  and NaOH fractions rich), with an  
562 intermediate level of P recovered in shoots and roots; (iii) groups 3-4 : Centr\_1, FFMSW\_1,  
563 FFMSW\_2, Centr\_2, Agri\_2 (intermediate to low total P-ARF); (iv) groups 5-7 : BW\_1, D1, D2 and  
564 BD (high total P-ARF and highest inorganic  $H_2O-P$ ); (v) group 6 : Agri\_1, Agri\_3, fibrous digestates.  
565 Agri\_1 has high P total recovery in both shoots and roots as BD, but Agri\_3 shows a high recovery  
566 only in roots. It seems that the fibrous characteristic enhances P recovery above all in in plants roots  
567 through the organic fraction of NaOH-P.

568 The groups obtained were similar to digestate typology groups, except for Agri\_2 and BD which were  
569 not in a same group. When doing statistical treatments on the shoots results and P speciation for the 8  
570 digestates used in addition to the 3 digestates used by Grigatti et al. (2019), some correlations were  
571 found. Water-P fraction was positively correlated with shoots ( $r=0.49$ ,  $p=0.09$ ). This result was also  
572 observed by Grigatti et al. (2017) for composts. As reported by Ahmad et al., 2018, available P consists  
573 in the exchangeable and soluble P corresponding to water and Na-bounded P fractions ( $H_2O+ NaHCO_3$ )  
574 whereas occluded P consists into P bound with metals (Fe, Al) and Ca. However, a simple linear model  
575 did not fit between inorganic  $H_2O-P$  fraction ( $H_2O-P_i$ ) and shoots P-ARF. PLS model was used showing  
576 that two main significant variables were needed to predict shoots P-ARF (Equation 10).  $H_2O-P_i$  fraction

577 impacted positively shoots P-ARF, as expected, whereas organic H<sub>2</sub>O-P fraction (H<sub>2</sub>O-P<sub>o</sub>) had a negative  
578 impact. Therefore, available form of P for shoots growth was associated to H<sub>2</sub>O-P<sub>i</sub> fraction, as expected.  
579 H<sub>2</sub>O-P<sub>o</sub> fraction is accessible but has to be mineralized to be available.

$$580 \text{Shoots}_P = 0.1153 \times \text{H}_2\text{O P}_i - 0.2777 \times \text{H}_2\text{O P}_o + 0.0249, r^2=0.71 \quad \text{Equation 10}$$

581 No correlations were found between ARF results and TP or phosphates concentrations ( $r=-0.12$ ,  
582  $p=0.74$ ,  $r=-0.26$ ,  $p=0.46$  respectively) meaning that the TP concentration is not enough to predict P  
583 uptake by plants.

584 Organic NaHCO<sub>3</sub>-P fraction (NaHCO<sub>3</sub>-P<sub>o</sub>) was positively correlated with roots ( $r=0.62$ ,  $p=0.025$  with  
585 D1, D2 and BD). Organic NaOH-P fraction (NaOH-P<sub>o</sub>) was also positively correlated with roots  
586 ( $r=0.79$ ,  $p=0.006$ ). A PLS regression model was found between roots P-ARF and inorganic and  
587 organic P content in NaHCO<sub>3</sub>-P and NaOH-P fractions (Equation 11).

$$588 \text{Roots}_P = 0.0955 \times \text{NaHCO}_3\text{P}_o + 0.0955 \times \text{NaOH P}_o - 0.0584 \times \text{NaHCO}_3\text{P}_i + 0.0128, r^2=0.8641$$

589 Equation 11

590 Negative impact of inorganic labile NaHCO<sub>3</sub> was found whereas organic P forms impacted positively  
591 the roots P-ARF. As previously mentioned, phytate and others phosphates esters can be extracted in  
592 the NaOH-P fraction (He et al., 2010). Roots can exude phosphatases able to hydrolyse phytate and  
593 organic P forms able to be absorbed by roots (Lambers et al., 2006; Gerke et al., 2015).

594 Interestingly, roots and shoots P recoveries were correlated with different P fractions. Finally,  
595 chemical nutrient equivalents were calculated for all the treatments. The obtained values were above  
596 100% except for Agri\_2 (35%) and Agri\_1 if only shoots were considered. That means that all the  
597 studied digestates (except from pig slurry) can substitute chemical P needs.

598 Similarly to N results, phase separation impacted the P fertilizing potential. The solid phase of  
599 centralised digestate Centr\_1 was more suitable as P fertilizer than Centr\_2. Composting impacted  
600 also the fertilizing value of the municipal waste digestates by lowering the P fertilizer potential relative  
601 to its respective digestate. This trend was also showed by Sludge\_1 (digestate) versus Sludge\_2  
602 (composted digestate). Knowledge of the availability of P and its effects makes possible to anticipate

603 fertilisation according to the soil composition (Albuquerque et al., 2012) and not only based on overall  
604 P analysis. Indeed, this was the case of sludge digestates Sludge\_1 and 2 which had the highest P  
605 contents (same level of Centr\_1) but their treatments obtained moderate P-ARF. From P results, first  
606 guidelines can be given according to the digestate feedstock typology for the studied calcareous soil  
607 and ryegrass. Pig manure digestate (Agri\_2) seemed not suitable for P fertilization. Fibrous digestates  
608 rich on wheat straw Agri\_1 and 3 had very low P fertilizing potential whereas FFMSW\_1 presented  
609 higher potential as the solid phase Centr\_1. The compost FFMSW\_2 had also low P fertilizing  
610 potential and the liquid phase Centr\_2 was intermediate. Sludge 1 and 2 had intermediary results and  
611 biowaste digestate BW\_1 showed high N and P fertilizing potential.

#### 612 **4. Conclusions**

613 This study focused on different typologies of digestates classified according to their process and to  
614 their feedstock. In this context, both P and N speciations showed a wide accessibility range according  
615 to feedstocks characteristics. The chemical accessibility indicators described the nutrient availability for  
616 plants and allowed the digestates classification on N and P fertilizing potential basis. The N and P  
617 speciation impacted the results from incubations with bare soil as well as the apparent coefficients of  
618 the use of N and P by the plant for its growth. Depending on the tissue collected (shoot or root), the  
619 speciation variables having a significant impact were different for P and N, for the type of calcareous  
620 soil used. First models were found to predict P recovery in shoots and roots using P speciation.  
621 Furthermore, C: N ratio value was significant and could be used for shoots N-ARF prediction whereas  
622 mineral water extracted N could be used for roots N-ARF prediction. Thus, a more detailed knowledge  
623 of the digestates would allow more adequate control of fertilization. Moreover, composting and phase  
624 separation impacted the nutrient recovery and can be used as an actuator to propose different organic  
625 fertilizers type. In terms of perspectives, field trials on contrasted soils qualities for crops with  
626 contrasting nutrient needs should be carried out in order to offer a guide to fertilization by type of  
627 digestate. Finally, N and P speciation studied could be used in dynamic models to improve soil and  
628 plant model predictions for digestate use in agriculture.

629 **Acknowledgements**

630 This project is supported by the Agropolis Fondation under the reference ID 1502-302 through the  
631 « Investissements d'avenir » program (Labex Agro:ANR-10-LABX-0001-01) and is part of the EU-  
632 funded project AgreenskillsPlus. Authors acknowledge Antoine Haddon for his useful advices.

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734 Table 1: Definition and origins of digestate samples

Sample Name	Type	Scale	Anaerobic Digestion conditions: type (dry or liquid), feed mode (batch or continuous) and temperature	Digester Hydraulic retention time	Post-treatment	Substrate	Reference
<b>Agri_1</b>	digestate	Operating scale (farmer)	Dry batch mesophilic	60 days	-	Cow manure	This study
<b>Agri_2</b>	digestate	Operating scale (farmer)	Liquid continuous mesophilic	60 days	-	Pig manure co-digested with energetic crop and vegetable residues	
<b>Agri_3</b>	digestate	Lab scale	Dry batch mesophilic	100 days	-	wheat straw silage	
<b>Sludge_1</b>	Solid phase from digestate	Operating scale (private agency)	Liquid continuous mesophilic	20 days	Press filter	wastewater treatment sludge	
<b>Sludge_2</b>	compost of solid digestate	Operating scale (private agency)	Liquid continuous mesophilic	20 days	Press filter and composting	digestate of wastewater treatment sludge (1/3) and green wastes (2/3)	
<b>FFMSW_1</b>	digestate	Operating scale (private agency)	Dry continuous thermophilic	20 days	-	Fermentable fraction from municipal solid waste (FFMW)	
<b>FFMSW_2</b>	compost of digestate	Operating scale (private agency)	Dry continuous thermophilic 20	20 days	Composting	96%FFMSW, 50% green waste, 14% agro-industrial wastes	
<b>BW_1</b>	digestate	Operating scale (governmental)	Liquid continuous mesophilic	60 days	-	Biowastes (60%) from supermarkets co-digested with agro-industrial wastes (28%) and crop residues (12%)	
<b>Centr_1</b>	Solid phase of digestate	Operating scale (governmental)	Liquid continuous mesophilic	45 days	screw press	Oil (20%), crop residues (10%), agro_industrial wastes (55%), sewage sludge (10%), biowaste (5%)	
<b>Centr_2</b>	Liquid phase of digestate	Operating scale (governmental)	Liquid continuous mesophilic	45 days	screw press		
<b>D1</b>	Digestate	Operating scale	Liquid continuous thermophilic	na	-	Wastewater treatment sludge	Grigatti et al. (2019)
<b>D2</b>	Digestate	Operating scale	Liquid continuous mesophilic	na	-	Wine sludge	
<b>BD</b>	digestate	Operating scale	Liquid continuous thermophilic	na	-	Bovine slurry and energy crops	

735

736

737 Table 2: Physico-chemical characteristics of the investigated digestates

Parameters	Units	Agri_1	Agri_2	Agri_3	Sludge_1	Sludge_2	FFMSW_1	FFMSW_2	BW_1	Centr_1	Centr_2	D1	D2	BD
<b>TS</b>	%	17.1%	4.4%	14%	22.4%	59.1%	19.7%	53.3%	24.9%	26.4%	6%	3.94%	3.09%	5.05%
<b>VS</b>	%TS	70.2%	70.1%	87.0%	51.7%	48.5%	48.7%	41.8%	79.0%	82.2%	60.0%	58.2%	59.4%	68.3%
<b>TOC</b>	$g.kg^{-1}$	382.90	404.71	454.83	283.35	258.01	279.30	232.55	438.64	438.17	320.53	344.2	397.8	515.0
<b>COD</b>	$g.kg^{-1}$	1104.00	1400.56	1235.50	679.00	726.00	712.09	536.00	1291.00	1245.00	1497.58	nd*	nd	Nd
<b>TKN**</b>	$g.kg^{-1}$	25.85	115.00	nd	40.36	26.40	22.20	13.61	29.79	23.33	75.67	nd	nd	nd
<b>TKN***</b>	$g.kg^{-1}$	27.98	<b>44.53</b>	17.72	43.06	26.61	17.78	15.39	30.89	28.62	<b>45.47</b>	47.8	44.9	43.0
<b>C/N</b>		13.68	3.52	25.67	6.58	9.70	15.71	15.11	14.20	15.31	7.05	7.2	8.9	12.0
<b>NH<sub>4</sub><sup>+</sup>-N**</b>	$g.kg^{-1}$	6.28	87.85	2.99	10.80	0.92	10.5	0.04	27.68	6.81	34.74	nd	nd	nd
<b>P</b>	$g.kg^{-1}$	5.36	10.37	4.00	20.44	16.15	4.21	4.23	10.79	6.93	17.98	7.1	18.4	6.2
<b>K</b>	$g.kg^{-1}$	21.22	27.41	12.98	2.15	4.90	11.49	8.22	7.25	4.86	16.10			
<b>S</b>	$g.kg^{-1}$	3.59	6.23	1.63	6.23	5.33	2.22	5.00	2.16	6.27	7.58		nd*	
<b>Al</b>	$g.kg^{-1}$	2.31	3.23	0.65	6.66	12.56	8.68	2.73	8.46	2.68	9.33			
<b>Ca</b>	$g.kg^{-1}$	18.53	23.08	13.44	69.08	41.25	37.05	29.72	42.31	17.56	27.08	10	37	11
<b>Fe</b>	$g.kg^{-1}$	1.58	2.81	0.94	42.60	37.48	5.79	5.06	7.89	11.54	12.07	1.9	8.6	1.0
<b>Ca/P</b>		0.29	0.27	0.24	2.08	2.32	1.38	1.19	0.73	1.67	0.67	1.4	2.0	1.7
<b>Mg</b>	$g.kg^{-1}$	5.49	6.28	1.56	3.52	3.72	3.24	4.29	3.47	1.98	5.13	2.8	11.6	5.2
<b>References</b>		This study										Grigatti et al. (2019)		

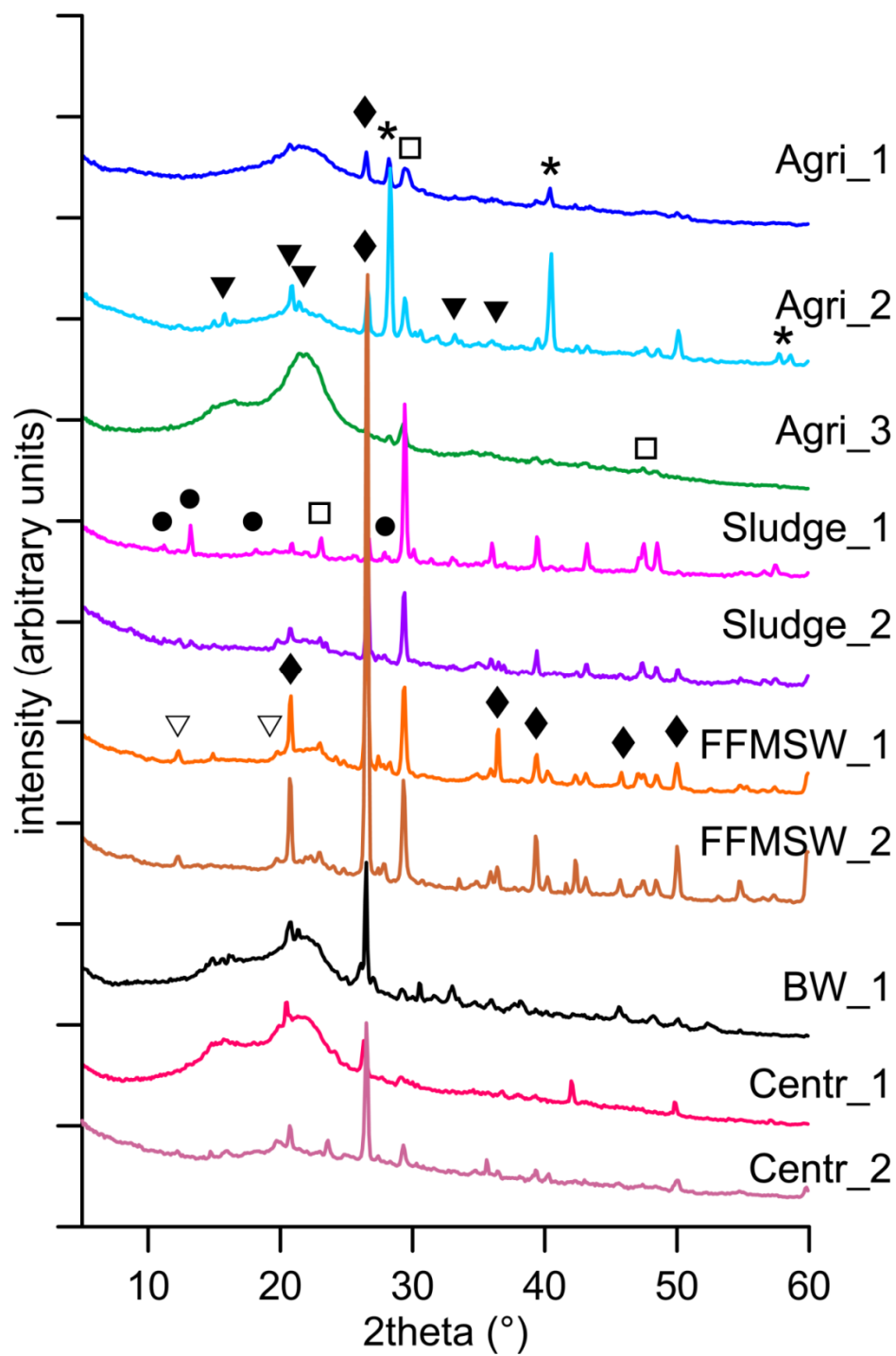
738 \* nd : Not determined

739 \*\* Measured on raw samples

740 \*\*\* Measured on from freeze-dried samples

741 Table 3: Dry biomass (DW), total P uptake and N uptake in ryegrass shoots during three successive  
 742 harvests (day 28-84) and roots at the final harvest (day 84)

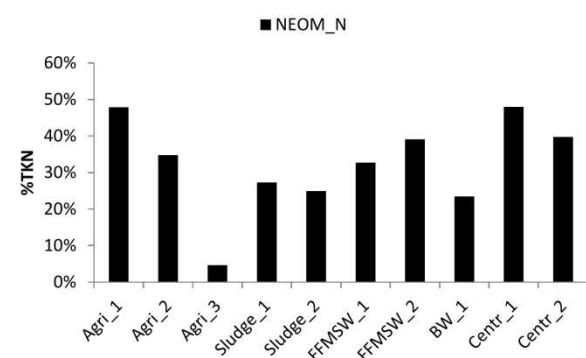
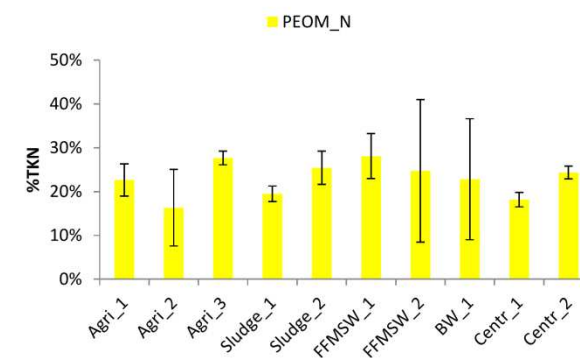
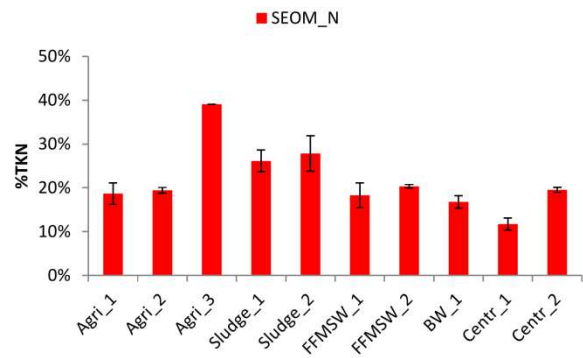
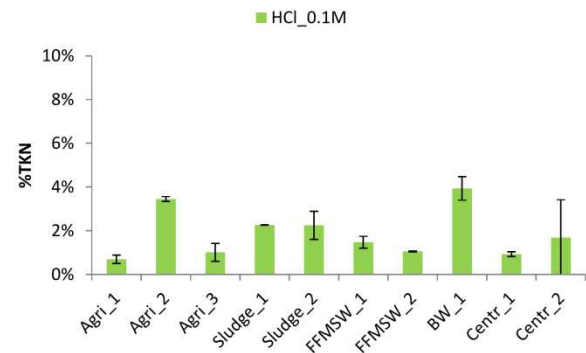
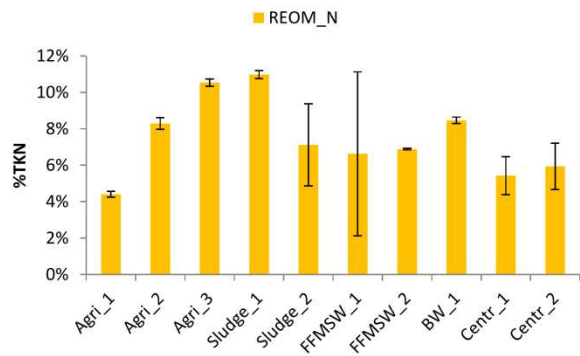
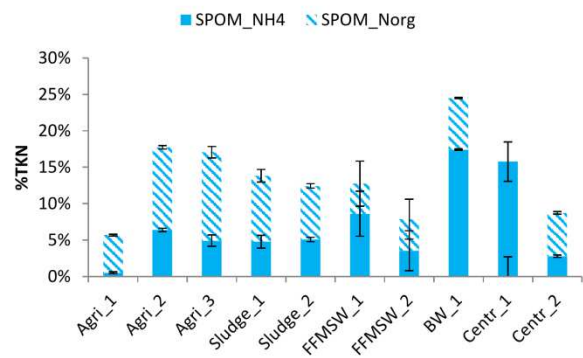
	Treatment	Days after sowing						Total
		Shoots				Roots	84	
		28	56	84	Mean	0-84		
	<i>DW (g pot<sup>-1</sup>)</i>							
	Ctrl-	0.87	0.53	0.25	0.55	1.65	1.25	2.90
	Ctrl+	1.00	0.70	0.39	0.70	2.09	1.56	3.65
1	Agri_1	0.88	0.50	0.29	0.56	1.67	1.51	3.18
2	Agri_2	0.86	0.58	0.35	0.60	1.79	1.23	3.02
3	Agri_3	0.84	0.44	0.27	0.52	1.56	1.82	3.38
4	Sludge_1	0.94	0.58	0.42	0.65	1.94	1.55	3.49
5	Sludge_2	0.94	0.57	0.37	0.63	1.88	1.87	3.75
6	FFMSW_1	0.85	0.58	0.36	0.60	1.79	1.91	3.70
7	FFMSW_2	0.88	0.47	0.26	0.54	1.61	1.32	2.93
8	BW_1	0.99	0.53	0.41	0.64	1.92	1.79	3.72
9	Centr_1	0.89	0.54	0.37	0.60	1.81	2.30	4.11
10	Centr_2	0.96	0.58	0.41	0.65	1.95	1.28	3.23
	<i>P uptake (mg pot<sup>-1</sup>)</i>							
	Ctrl-	1.93	1.60	1.02	1.52	4.55	2.25	6.80
	Ctrl+	2.17	1.48	1.43	1.70	5.09	2.24	7.33
1	Agri_1	2.41	1.14	1.61	1.72	5.16	2.87	8.03
2	Agri_2	1.81	1.15	1.76	1.58	4.73	2.19	6.92
3	Agri_3	1.87	1.07	1.76	1.57	4.70	2.86	7.56
4	Sludge_1	2.28	1.23	2.70	2.07	6.21	2.96	9.17
5	Sludge_2	2.29	1.53	2.32	2.05	6.14	3.18	9.32
6	FFMSW_1	2.05	1.76	2.41	2.07	6.21	2.62	8.84
7	FFMSW_2	2.65	1.13	1.35	1.71	5.13	2.22	7.35
8	BW_1	2.76	1.40	2.38	2.18	6.54	2.39	8.93
9	Centr_1	2.77	1.36	2.26	2.13	6.39	2.58	8.97
10	Centr_2	2.67	1.53	2.31	2.17	6.51	2.25	8.76
	<i>N (mg pot<sup>-1</sup>)</i>							
	Ctrl-	25.01	9.32	6.52	13.62	40.85	12.44	53.28
	Ctrl+	45.70	16.69	9.23	23.87	71.62	14.89	86.51
1	Agri_1	25.29	7.35	8.21	13.62	40.85	14.76	55.61
2	Agri_2	29.41	9.03	8.98	15.81	47.42	13.31	60.73
3	Agri_3	21.84	6.48	6.80	11.70	35.11	14.71	49.82
4	Sludge_1	36.91	10.51	12.39	19.94	59.81	15.13	74.93
5	Sludge_2	35.32	9.72	10.52	18.52	55.55	17.49	73.05
6	FFMSW_1	28.77	10.34	9.67	16.26	48.78	15.35	64.13
7	FFMSW_2	29.80	8.97	7.41	15.39	46.17	15.45	61.62
8	BW_1	32.03	9.97	11.88	17.96	53.88	16.51	70.39
9	Centr_1	30.74	9.28	10.00	16.67	50.02	18.45	68.47
10	Centr_2	34.63	10.39	12.16	19.06	57.18	14.04	71.22



743

744 Figure 1: X-ray diffraction of the digestates. The main peaks of different crystalline phases are

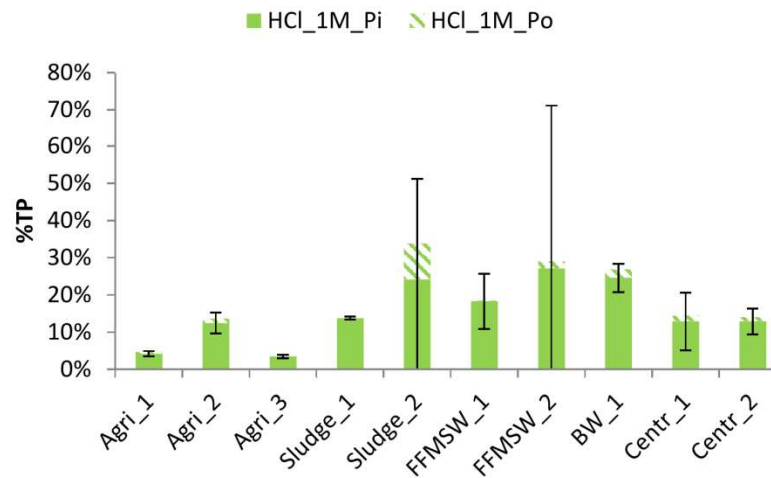
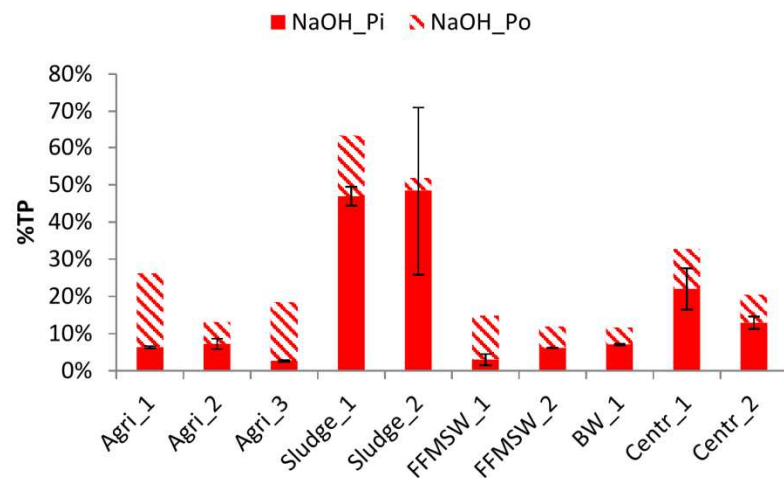
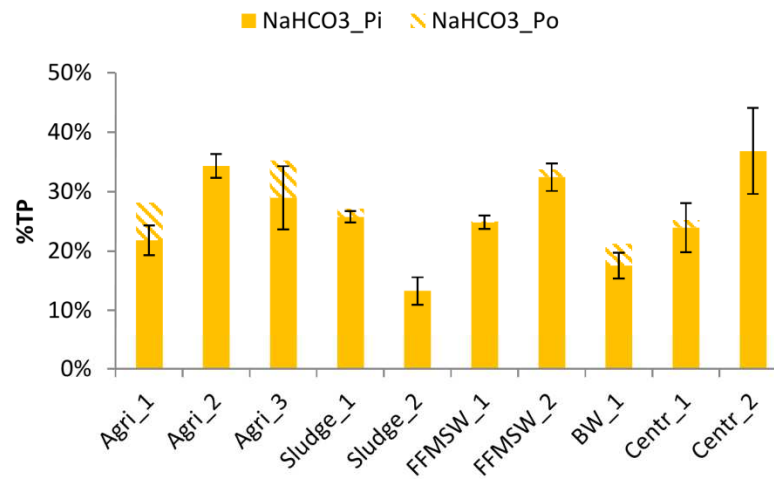
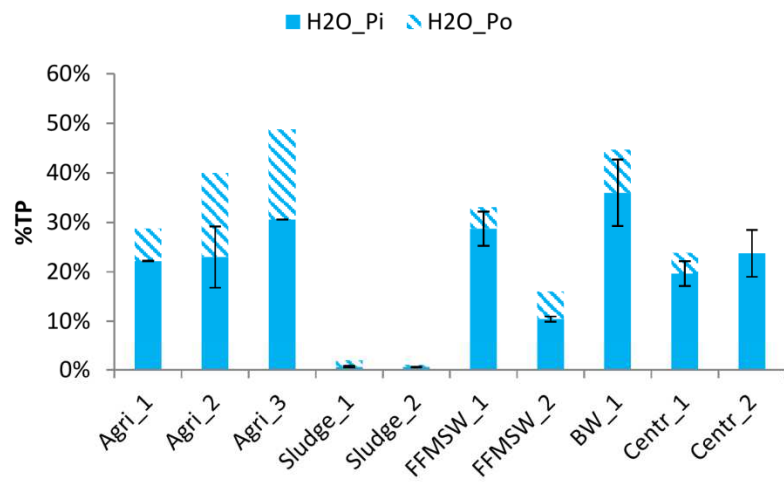
745 identified by symbols: ▼ struvite; ◆ quartz; □ calcite; ● vivanite; \* sylvine; ▽ kaolinite.



746

747 (a)

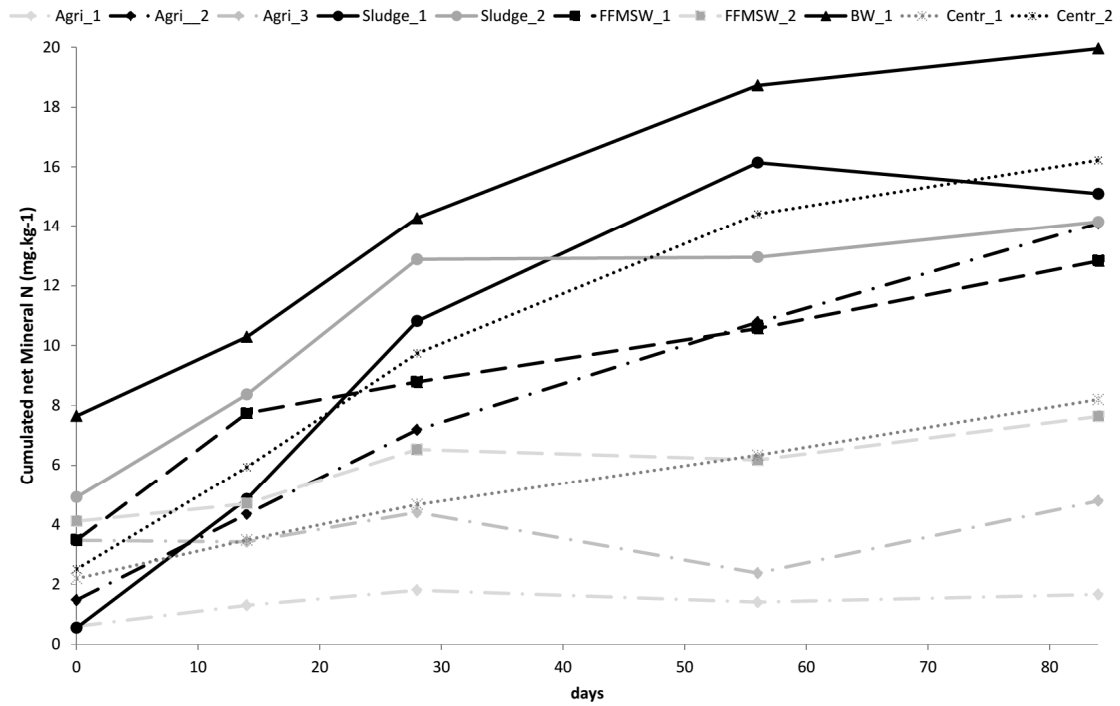
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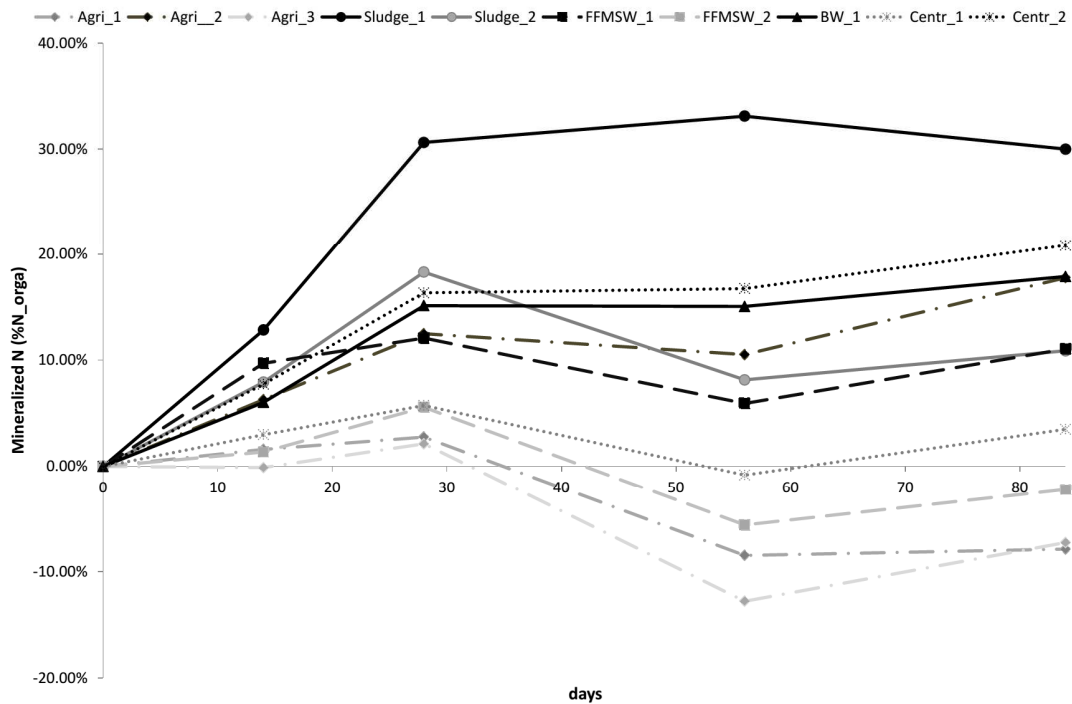
(b)

750 Figure 2: SCE fractions of TKN (a) and TP (b) obtained for all the digestates, according chemical accessibility. Error bars, SE (n=2)



751

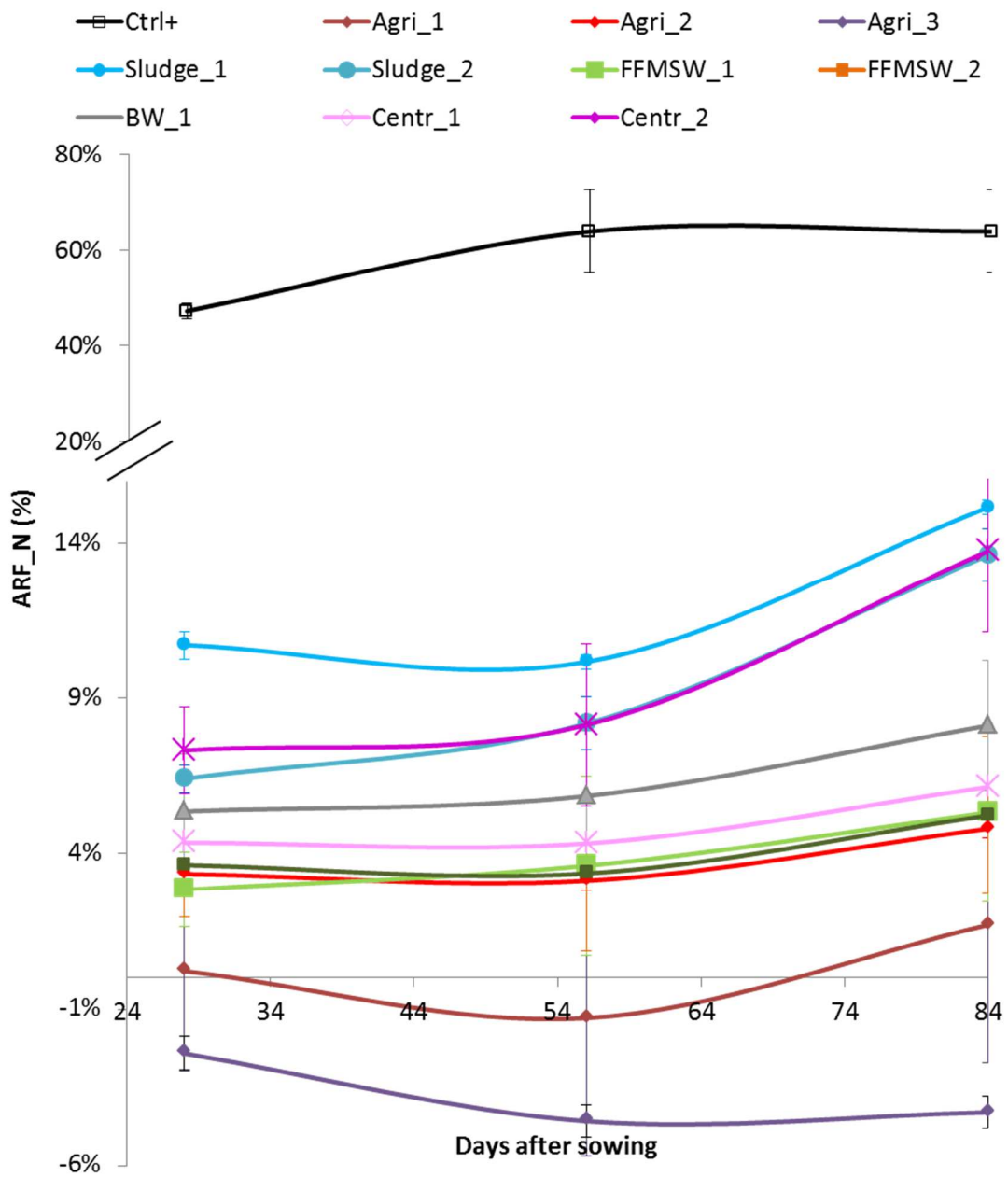
752 (a)



753

754 (b)

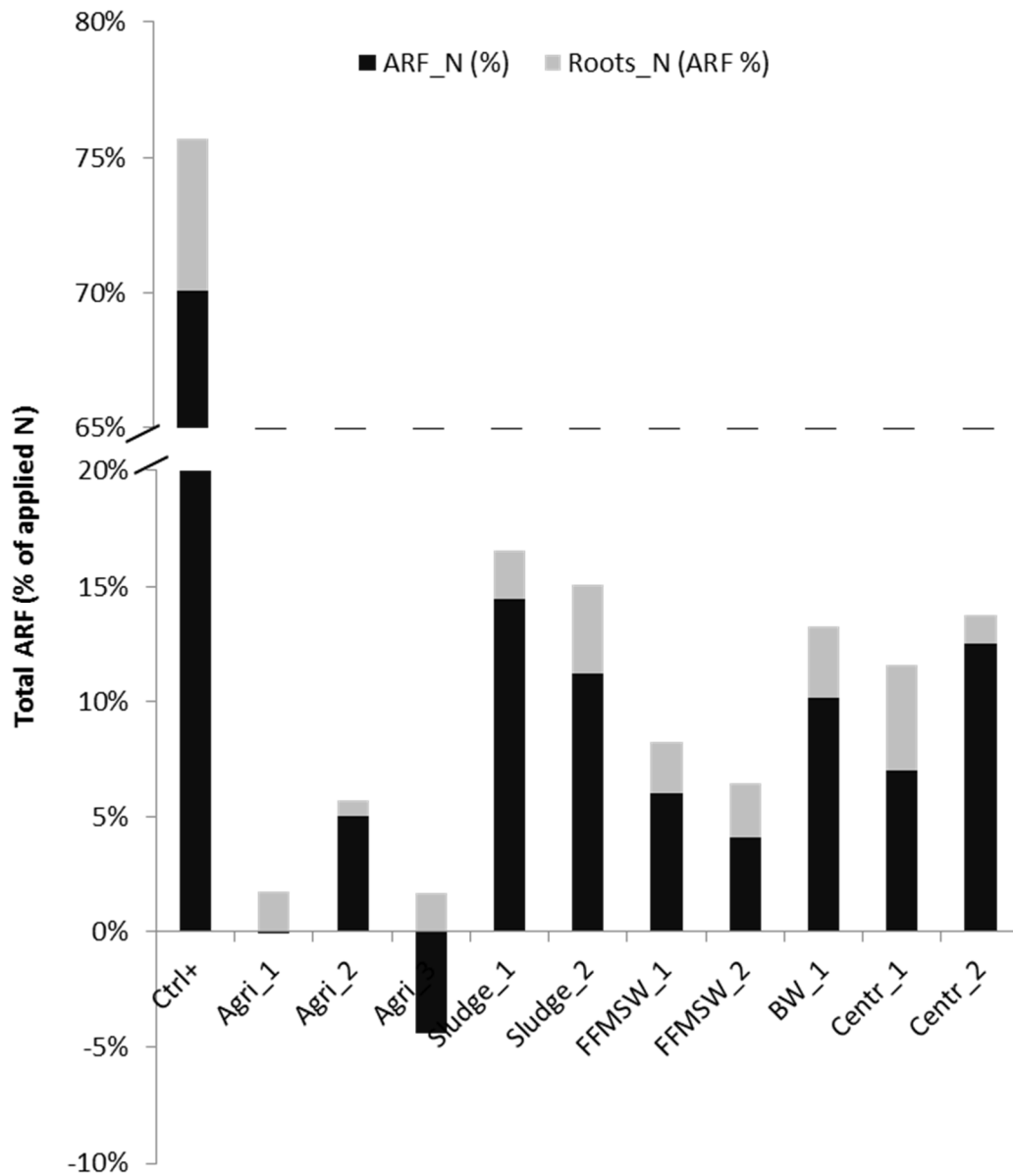
755 Figure 3: Cumulated mineral nitrogen concentration evolution (a) and cumulated mineralized organic  
 756 nitrogen percentage (b) during soil incubation. Net values. *Black: high mineralized N group; Grey:*  
 757 *low mineralized N group (circle+full line: sludge digestates, square+large dashes: FFMSW*  
 758 *digestates, diamond+ dashes and dots line: Agrowastes digestates, triangle: biowaste digestate,*  
 759 *cross+dotted line: centralised digestates)*



760

761 (a)

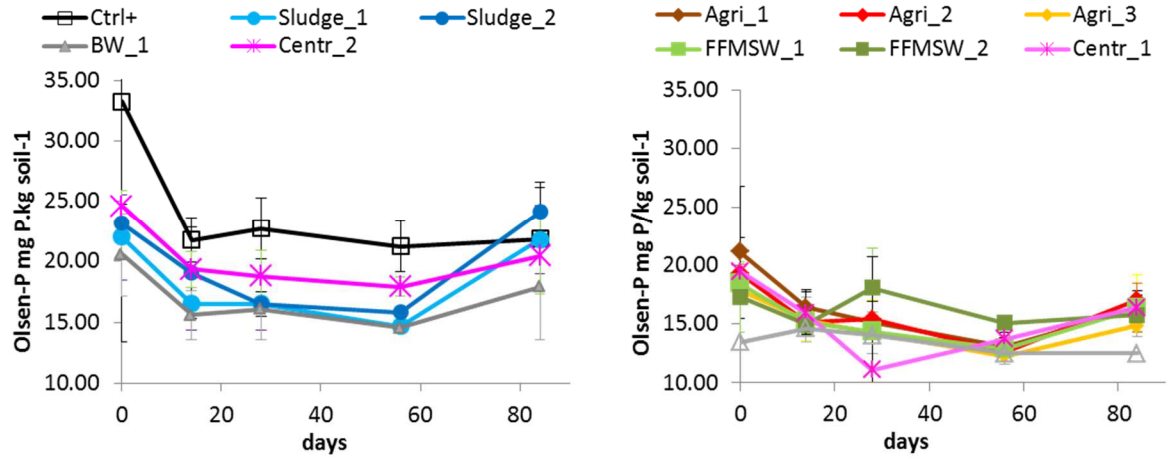
(a)



762

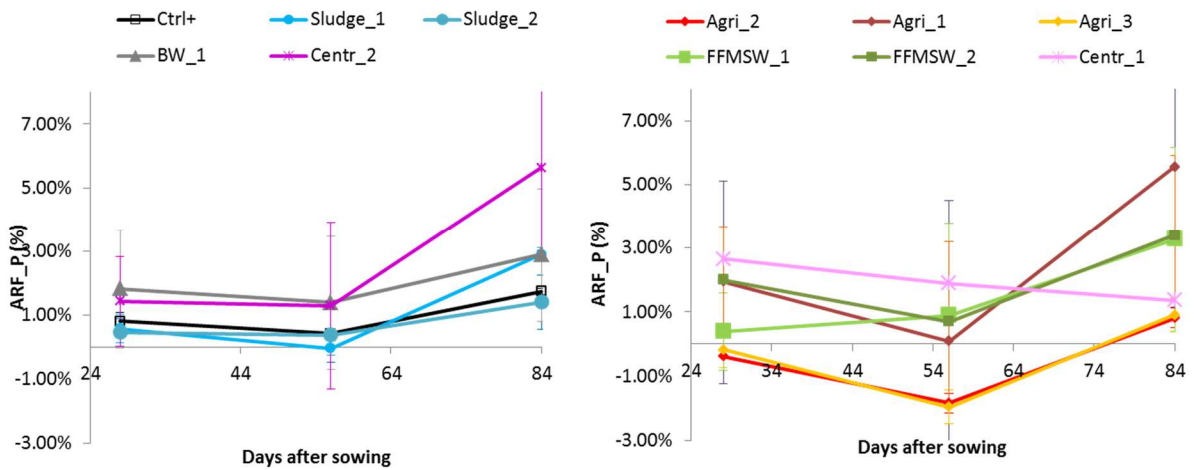
(b)

763 Figure 4: Nitrogen apparent recovery fraction (ARF) of ryegrass shoots (a) and in both shoots and  
 764 roots (b) in three harvests during pot trial. Error bars, SE (n=3)



765

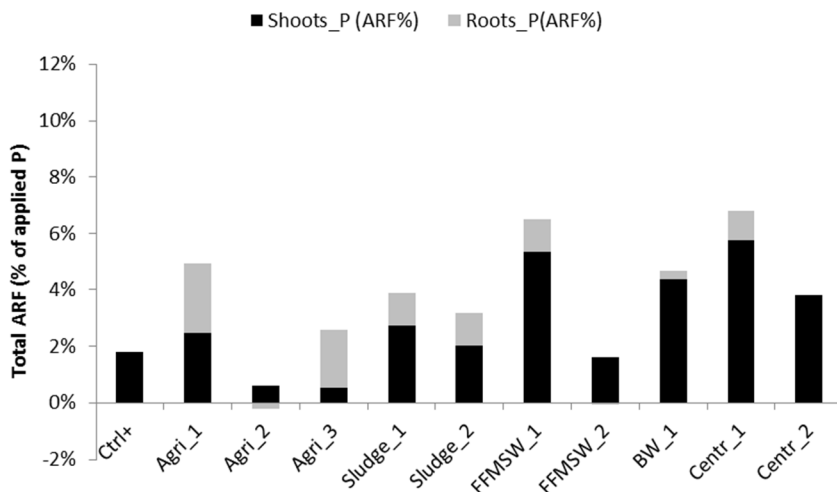
766 (a)



767

768

(b)



769

(c)

770 Figure 5: Olsen-P evolutions during the soil incubation of the tested digestates (a), cumulative  
 771 phosphorous apparent recovery fraction (ARF) of ryegrass shoots (b) and in both shoots and roots (c)  
 772 in three harvests during pot trial. Error bars, SE (n=3)