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# Long-term, low technicality sewage sludge amendment and irrigation with treated wastewater under Mediterranean climate: impact on agronomical soil quality

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# 1 Long term, low technicality sewage sludge amendment and irrigation with treated 2 wastewater under Mediterranean climate: impact on agronomical soil quality.

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## 9 10 Abstract

11  
12 We evaluated the agronomic quality of loam to clay-loam soils from a Mediterranean area, that have been  
13 submitted for more than 10 years to irregular and poorly controlled treated wastewater (TWW) irrigation and  
14 sewage sludge (SS) amendment, both TWW and SS issuing from a domestic effluent treatment plant. A soil was  
15 submitted to SS-amendment only, another to TWW-irrigation only, another to both treatments and another was  
16 not submitted to any of these treatments. We found that the agronomic quality of the treated soils has not  
17 decreased. In treated soils it was observed a slight increase of pH, an increase of the CEC, no change of either  
18 salinity or electrical conductivity. *SAR* and *ESP* remained satisfactory, below 13 and 15, respectively, and no  
19 infiltration problems were observed, indicating that rainwater percolation was sufficient to leach the salt brought  
20 by SS or TWW. The soil organic matter (SOM) increased in all SS-amended and/or TWW-irrigated soils, the  
21 increase being higher in SS-amended soils. The SOM kept a satisfactory C/N ratio, lower than 15; the SUVA  
22 and E2/E3 index showed normal characteristics of the water-extracted organic matter (WEOC). Both SS  
23 amendment and TWW irrigation brought high amounts of nutrients in the considered soils and increased the  
24 nutrients disponibility, especially regarding K and P, as shown by the nutrient concentrations in the soil water  
25 extracts. The pH increase does not seem to be a problem for the bioavailability of trace elements, at least  
26 regarding Cu, Zn, Co and Fe. Complementary studies should be undertaken for Ni. These observations highlight  
27 the potential agronomical benefits of application of wastewater and sewage sludge, even without great  
28 technicality, the better results being obtained using both SS amendment and TWW irrigation.



29

1  
2 30 **Keywords:** Wastewater reuse, sewage sludge reuse, Mediterranean zones, soil quality, water-extractible  
3  
4 31 nutrients, metal bioavailability  
5

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7 32

8  
9 33 **Acknowledgments**

10  
11 34 We thank the Algerian Ministry of High Education and Scientific Research (MESRS) and the Mouloud  
12  
13 35 Mammeri University for the doctoral scholarship and facilities that allowed this work.  
14  
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17  
18  
19 37 **Introduction**

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24 39 In Algeria, the quantity of sewage sludge (SS) currently produced from wastewater treatment plants is  
25  
26 40 around 250 000 tons per year; it is estimated that the quantities of sludge will reach 450 000 tons in 2020. The  
27  
28 41 treatment techniques applied for sludge are incineration, landfill, composting or spreading on agricultural land. A  
29  
30 42 large part of the sludge is deposited along rivers or in domestic waste disposal sites, while only 25% are spread  
31  
32 43 on agricultural lands.  
33

34 44 Moreover, the volume of treated wastewater (TWW) in Algeria is estimated at 560 000 cubic meters and  
35  
36 45 65% of this water are used for the irrigation of cultivated lands (Hannachi et al. 2014). The scarcity of water  
37  
38 46 resources has increasingly encouraged recycled wastewater as suitable resource substitute (Pereira et al. 2002;  
39  
40 47 Qadir et al. 2010) since this practice offers various advantages: the reuse of TWW avoids their direct discharge  
41  
42 48 into streams, supports fresh water resources protection, recycles nutrients and minimizes water and soil pollution  
43  
44 49 with regard to uncontrolled disposal (Rehman et al. 2013). However, wastewater irrigation also involves  
45  
46 50 potential risks to soil quality and to the quality of crops production, because of the presence of various inorganic  
47  
48 51 substances, including potential toxic elements (Cr, Cd, Pb, Ni and Hg) (Rehman, et al. 2013; Elifantz, et al.  
49  
50 52 2011), and to high levels of Na, which in the long term could degrade the physical properties of the soils (e.g.  
51  
52 53 Halliwell et al. 2001; Stevens et al. 2003). These risks must be taken into account and evaluated.  
53  
54

55 54 The sludge spreading practice on agricultural lands is considered as the easiest and economically cheapest  
56  
57 55 disposal procedure (Pedrero et al 2010; Fytili and Zabaniotou 2008). As generally admitted wastewater and  
58  
59 56 sludge are attractive alternatives for water and fertilizers because they provide the nutrients and water necessary  
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57 for crop growth. Application of unprocessed wastewater sludge and wastewater sludge have been reported as  
58 beneficial organic fertilizers (Singh and Agrawal 2008; Roca-Perez et al. 2009). Wastewater irrigation and  
59 sludge spreading lead to higher yields than usual for most crops and reduce the requirements for fertilizers,  
60 resulting in net cost savings for farmers (Warman and Termeer 2005; Zebarth et al. 2000; Hussain et al. 2002).  
61 The sludge is a source of major nutrients such as P, K, S, Ca, Mg, micro nutrients and organic matter. Sludge  
62 applications usually improve the physico-chemical and biological soil properties, such as porosity, field capacity  
63 pH, organic matter content, available phosphorus (Mtshali et al. 2014; Delibacak et al. 2009; Tarchouna et al.  
64 2010). For these reasons, the sludge is considered as an interesting alternative as fertilizer in agricultural lands  
65 and as the tool to maintain the soil carbon stock. On the other hand, sludge produced after wastewater treatment  
66 are particularly difficult to manage due to the increasing quantities produced and the potential pathogenic  
67 organisms and metal contents present in the sludge. The great variability in the sludge composition requires a  
68 specific analysis before use.

69 In Algeria, the application of sludge and waste water on agricultural land has been developed only recently  
70 and is spreading rapidly. The research and scientific contributions that address the above issues are scarce and  
71 only considered the agronomic value of sludges (Ramdani et al 2015; Benmouffouk 1994; Igoud 2001). Our  
72 informal discussions have shown that local farmers in Algeria have observed a significant increase in yields after  
73 using sludge or TWW, but no data are available.

74 In the absence of regulatory requirements in Algeria and as the agricultural use of SS and TWW began  
75 empirically, without prior local agronomic experiment and without precise control of the quantities brought, our  
76 objective is to verify that the existing recovery routes for sewage sludge and TWW are both economically viable  
77 on the long-term and safe in terms of health. Planning efforts toward this goal should include the evaluation of  
78 potential changes in soil quality when amended with SS or irrigated with TWW and the understanding of the  
79 related agronomic and environmental issues.

80 This part of the study is focused on the impact of 10-years wastewater irrigation and sludge amendments on  
81 soil agronomic quality, in a context where the application of SS amendment or TWW irrigation is irregular and  
82 relatively poorly controlled. The potential contamination with potentially toxic elements will be considered in a  
83 further publication.

84



## 85 Study Site

86

87 The study area is located in northern Algeria, in the Corso city territory (district of Boumerdes). The  
88 geological substrate is an upper Pliocene compact red sand that recovers an older marl. The region climate is  
89 Mediterranean, characterized by hot and dry summer followed by a relatively cold and rainy season, from  
90 November to May, with an annual average rainfall of 650mm.

91 The use of sewage SS and TWW issued from the Boumerdes wastewater plant had begun in 2002 on  
92 agricultural land cultivated for table grape (Dattier de Beyrouth variety). The sludge is spread once a year at an  
93 estimated dose between 15 and 20 tons per hectare, while the drop-irrigation with TWW is practiced from May  
94 to October to compensate for the hydric shortages.

95 A specific pipeline network transports daily 5200m<sup>3</sup> of TWW for irrigation of 70 hectares. It was very  
96 difficult to obtain reliable information about the use of fertilizer in the selected farms. Commonly used fertilizers  
97 are ammonium nitrate NH<sub>4</sub>NO<sub>3</sub> 33.5% N; urea 46% N; sulphate of potash K<sub>2</sub>SO<sub>4</sub> 50% K<sub>2</sub>O and superphosphate  
98 Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>.CaSO<sub>4</sub> 20% P<sub>2</sub>O<sub>5</sub>. Unfortunately, applied quantities remain unknown by the farmers themselves.

99 We sampled five representative vineyards soils, similar in terms of parent rock, soil type, topographical  
100 situation and technical farming. They differentiate only for the use of SS, TWW and river water applications. The  
101 map (Fig.1) locates the sampling sites, situated about 1km from the sea coast. The different treatments to the soil  
102 surface have been applied for 14 years and the samples collected were representative of each of them. We  
103 collected a soil receiving sewage sludge (S-SS), a soil irrigated with TWW (S-TW), a soil receiving both sludge  
104 and TWW (S-STW), and a reference soil that did not received neither sludge or TWW (S-NA). Some farms use  
105 river water to irrigate vineyards in summer; so we also sampled a farm (S-WR) that have been using for 5 years  
106 river water issued from the Corso River, which is issued from the Keddara dam, in order to compare with sludge  
107 and TWW applications. Unlike the other studied soils, however, the S-WR soil received organic amendments of  
108 cattle manure.

109 The sewage sludge (SS) and the treated wastewater (TWW) were sampled at the Boumerdes wastewater  
110 Treatment Plant.

111



## 112 **Materials and methods**

### 114 **Soil Sampling**

116 The sampling was performed in April 2013 at three depths (P1: 0-10cm; P2: 10-25cm; P3: 25-40cm). The  
117 soil samples were taken during the same month in order to take into account the turn-over process that can lead  
118 to carbon content variability. At each site, and for each depth, five cores were sampled at the corners and at the  
119 center of a 1m square plot of 60cm deep with an auger. A composite soil sample was generated by thoroughly  
120 mixing together the five subsamples from a given depth. Samples were air-dried and sieved at 2 mm. Aliquots of  
121 the <2 mm soil fraction were ground in an agate mortar and stored in polyethylene containers at 4°C until  
122 analysis. The dry bulk density ( $\rho_d$ ) was determined using the ring method.

### 124 **Analysis**

126 All solutions were prepared with high purity water (resistivity  $\approx 18 \mu\text{s}\cdot\text{cm}^{-1}$ , Milli-Q Plus system, Millipore  
127 Corporation, USA) and analytical grade (Merck). For sludge and soil samples, pH was determined at a 1:5 soil-  
128 to-water ratio. The soil texture was determined with the Robinson pipet method. In the literature, water-  
129 extractable organic carbon or major species are measured on water-extracts obtained using a soil-to-water ratio  
130 ranging from 1:2 (Zsolnay 2003) to 1:10 or more (Zeng et al. 2015). Here we choose a 1:10 soil-to-water ratio,  
131 agitation for 24 h and filtration at 0.45  $\mu\text{m}$ . Organic carbon and total nitrogen of solid samples (soils and SS)  
132 were determined using a CHNS apparatus (Shimadzu, Flash2000) after  $\text{H}_3\text{PO}_4$  pretreatment (Pastor et al. 2011).  
133 For liquid samples (TWW and water extracts) dissolved organic carbon was determined using a TOC analyser  
134 (TOC-V CSH, Shimadzu) after acidification and nitrogen purging. At least three sample replicates and three  
135 analysis replicates for each sample were performed in order to meet statistical acceptance requirements. The soil  
136 organic matter (SOM) was calculated from the soil organic carbon (SOC) using the formula:

$$137 \text{ SOM (\%)} = \text{SOC (\%)} \times 1.72$$

138 Cation exchange capacity (CEC) was determined for sludge and soil samples by the cobaltihexamine  
139 procedure (Tarchouna et al. 2010). Metal contents were quantified by ICP-AES (inductively coupled plasma



140 atomic emission spectrophotometry) while ions concentrations in solution (nutrients and other major species)  
141 were determined by ionic chromatography (Dionex-DX-120). The electrical conductivity EC was determined on  
142 a 1:10 soil-to-water ratio using a conductimeter. In order to compare with the literature data, we calculated the  
143 EC corresponding to saturated paste extract ( $EC_{SP}$ ) from the value measured on the 1:10 extract ( $EC_{1:10}$ ) using  
144 the following correlation equation (de Souza et al. 2013) where  $EC_{SP}$  and  $EC_{1:10}$  are expressed in  $mS\ cm^{-1}$  :

$$EC_{SP} = 23.654 EC_{1:10} - 0.2664$$

146 The exchangeable sodium percentage (*ESP*) was calculated using the CEC and the Na concentration in the  
147 1:10 soil extract. The sodium adsorption ratio (*SAR*), widely used to evaluate probable risks of irrigation water  
148 on the soil structure and the sodicity hazard of a soil, was calculated using the formula

$$SAR = \frac{[Na^+]}{\sqrt{\frac{[Ca^{2+}] + [Mg^{2+}]}{2}}}$$

151 where  $[Na^+]$ ,  $[Ca^{2+}]$  and  $[Mg^{2+}]$   $mmol_c\ L^{-1}$  concentrations in the 1:5 water extract (He et al. 2015).

152 The organic matter in the water extract (WEOC, water extractable organic matter) was characterized by  
153 UV-visible spectrometry to calculate the SUVA 254 index (UV absorbance at 254 nm normalized for dissolved  
154 organic carbon concentration, here expressed in  $L.mgC^{-1}\ m^{-1}$ ) and the E2/E3 ratio (ratio of UV absorbance at 250  
155 and 365 nm). The SUVA 254 index is indicative of the organic matter aromaticity, usually related to  
156 humification (Weishaar et al. 2003), and the E2/E3 ratio is positively related to low-molecular-weight  
157 compounds (Peuravouri and Pihlaja 1997).

158 Bioavailability of trace elements likely to cause deficiencies in soils with basic pH (Cu, Zn, Mn, Co, Ni) was  
159 evaluated by water- and sodium acetate extractions. Water-extraction was performed at a 1:5 soil-to-water ratio,  
160 agitation for 24 h and filtration at  $0.45\ \mu m$ . Sodium acetate extraction was performed by introducing 2 g soil or  
161 sludge in 20mL  $KNO_3$  1M, shaking for 16 h, adding 16mL of  $CH_3COONa$  1M, shaking 3h and filtrating at  
162  $0.2\ \mu m$ . Bioavailability of iron was evaluated by quantifying the amorphous iron phase by citrate/ascorbate  
163 extraction: 3g of soil was suspended in 60mL of a  $NaHCO_3$  0.024M,  $C_6H_9Na_3O_9$  0.02M,  $C_6H_8O_6$  0.003M  
164 solution adjusted to pH 8 by N purging, shaken for 24 h and filtrated at  $0.2\ \mu m$ . Metal contents were quantified  
165 by ICP-AES.



166 Principal components analysis (PCA) were performed using XLSTAT 2017 on various set of the obtained  
167 data. PCA project multidimensional data cloud to subspace such as maximize the residual variance, choosing the  
168 most relevant directions (principal components) in the data (Jolliffe 2002). We chose a correlation-type PCA on  
169 standardized variables to avoid sensitivity to the scaling of the variables.

## 171 **Results and discussion**

### 173 **Main agronomical characteristics of the studied soils, sludge and wastewater**

175 The Tables 1 and 2 gives the main agronomical characteristics of the soils, SS and TWW. Regarding soils,  
176 values given in Table 1 refer to the 0-40cm soil layer as they were calculated as the mean of the values obtained  
177 over the three sampled depths weighted by the thickness of the sampled layers.

178 The SS contained high contents of organic matter (OM) and total nitrogen, with a low C/N ratio (6.4). The  
179 SS water extract showed high concentrations of dissolved OM (3826 mg.L<sup>-1</sup>), but also of N (mainly as nitrites  
180 and ammonium, 25.4 and 9.2 mg kg<sup>-1</sup>, respectively, corresponding to 0.55 and 0.51 mmol kg<sup>-1</sup>, respectively), Ca,  
181 Mg, K and P. Its high SO<sub>4</sub><sup>2-</sup> value may be related to the possible use of ferric sulfate or chlorosulfate during the  
182 wastewater treatment process, which was expected with regard to their high solubility. The TWW brought those  
183 elements in the same relative proportions than the ones in SS water extract, but Na<sup>+</sup> and Cl<sup>-</sup> had relative higher  
184 values than in SS. The given values for nutrients confirm the potentially positive agronomical effect of spreading  
185 sludge or irrigating with TWW. We must ensure, however, that beneficial impacts are observed without negative  
186 consequences on soil quality parameters in order to maintain long-term high yields and environmental quality.  
187 The details of the composition of the soils depth by depth are given in Table 2.

188 *Granulometry and dry bulk density* – The five studied soils showed similar mineral particles size  
189 distribution. According to the texture triangle, S-SS is loam, S-TW and S-NA are silt-loam, S-STW and S-WR  
190 are clay loam, this variability being consistent with the natural variability of the red sand parent material. Values  
191 of dry bulk density are in the range expected for this type of soil. They remained lower in the topsoil than in  
192 depth, indicating that the use of SS or TWW did not lead to a collapse of the porosity of the surface horizons as  
193 frequently observed after such applications (Singh et al. 2008). Here no infiltration problems were related,  
194 However, attention should be paid since these observations, even if numerous, are not general. As observed in a





195 Mediterranean soil irrigated with TWW by Tarchouna et al (2010), saturated hydraulic conductivity remained  
196 high enough to allow water percolation during rainy periods and subsequent leaching of accumulated salts,  
197 preventing soil salinization.

198 Soil pH – All soils exhibited a neutral to slightly alkaline pH (USDA 1993): pH of all samples was  
199 comprised between 6.93 and 7.70. With regard to the reference S-NA soil, the application of SS, TWW and  
200 SS+TWW raised the average soil pH from 7.0 to 7.5, 7.2 and 7.6, respectively, which is slight but significant.  
201 Most studies showed for non-acidic soils that sludge application results in a pH decrease presumably due to  
202 humic substances decomposition or accelerated nitrification (Ahmed et al. 2010 ; Brallier et al. 1996). Each case,  
203 however, is dependent on the initial soil pH and the composition of the amendment. Here the high  $K^+$ ,  $Ca^{2+}$  and  
204  $Mg^{2+}$  basic cations input from SS and TWW combined to a low soil OM contents is likely to increase the soil  
205 alkalinity and subsequently the soil pH. In agricultural soils, pH should preferentially be between 6 and 7  
206 because higher values, especially  $>7.5$ , result in reduced availability of several nutrients or micronutrients,  
207 particularly P, Fe, Mn, Cu, Zn, Co and Ni (Jones 2012). Here the pH observed in soils SS-amended and/or  
208 TWW-irrigated remained compatible with good soil quality and crop production.

209 *Cation exchange capacity CEC* – CEC values are in the range usually observed for this type of soils. They  
210 were higher in SS-amended and/or TWW-irrigated soils than in the S-NA reference soil, which can be related to  
211 the lower clay and organic matter content of the latter. There is no significant change in CEC between SS-  
212 amended and/or TWW-irrigated soils that can be attributed to the type of amendment or irrigation.

213 *Electrical conductivity EC* – The EC value for TWW indicated a good quality with regard to salinity  
214 according to Fipps (1996). The  $EC_{1:10}$  values in all studied soils extracts remained below  $70 \mu S cm^{-1}$  and values  
215 were below or comparable to the values observed for the reference soil. The corresponding  $EC_{SP}$  values were  
216 lower than  $2 mS cm^{-1}$ , indicating a low salt rank and very little chance of injury on all plants (Lamond and  
217 Whitney 1992). It was observed in several studies that sludge application induces an EC increase (Clapp et al  
218 1986). Here the maintenance of low EC values in SS and STW soils despite the fact that SS extract  
219 exhibited significant conductivity ( $677 \mu S cm^{-1}$ ) indicates sufficient leaching by annual deep drainage of  
220 the soluble species brought in, especially  $Na^+$  and  $Cl^-$ .

221 *Sodium adsorption ratio (SAR) and exchangeable sodium percentage (ESP)* – Notwithstanding the fact that  
222 the TWW has a SAR around 15, all studied soils exhibited a SAR below 13 and a ESP below 15 (Table 1),



223 indicating good soil structural conditions (US Salinity Lab Staff 1954) and confirming a sufficient leaching of  
1 Na<sup>+</sup>.  
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3

4 *Organic matter (SOM)* – Because soil organic matter (SOM) can be associated with different soil chemical,  
5  
6 physical and biological processes, it has been widely considered as one of the best soil quality indicator. The  
7  
8 SOM impacts, among others, soil structural stability, nutrient cycling, water retention and microorganism  
9  
10 dynamics, which is related to SOM mineralization. Here the SOM content of the upper horizon is low (1.6%) in  
11  
12 the S-NA reference soil, as expected for a Mediterranean soil where high temperature and low rainfall favor  
13  
14 organic matter mineralization (Fantappiè et al. 2011). The higher value (4.0 %) in the S-SS topsoil can be related  
15  
16 to the high OM content of the sludge, as frequently observed (Haynes 1998; Gerzabek et al. 2001; Fernandez et  
17  
18 al. 2006; Triberti et al. 2016). The S-TW and S-STW topsoils showed intermediate values (2.2 and 2.8 %,  
19  
20 respectively). Although the S-STW soil received sludge, its lower SOM content compared to S-SS is likely  
21  
22 related to a priming effect due to TWW-enhanced mineralization of the sludge organic matter. The high SOM  
23  
24 content observed in the S-WR soil was directly related to cattle manure amendments. In all soils but S-WR, the  
25  
26 C/N ratio was lower than 15, indicating a potentially good release of N during SOM decomposition process. The  
27  
28 high C/N observed for the S-WR soil was likely related to wheat straw mixture with cattle manure.  
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31 *Water extractable fraction of SOC (WEOC)* – Although the WEOC is only a small percentage of SOC, it  
32  
33 has a critical role in soils properties. It is sensitive to soil management, easily bioavailable for microorganisms  
34  
35 and plants, and therefore a primary source of mineralizable C, N, P, and S, and exhibits a rapid turnover (minutes  
36  
37 to months) (Ćirić et al. 2015). Its greater mobility can impact the transfers of contaminants, especially heavy  
38  
39 metals. The relationships between WEOC and SOM are given in Fig. 2. The WEOC values ranged from 49 to  
40  
41 215 mg kg<sup>-1</sup>, in the range of values usually observed in cultivated soils (Chantigny 2003). A positive correlation  
42  
43 (R<sup>2</sup>=0.88) was observed between SOM and WEOC in SS-amended and/or TWW-irrigated soils. The S-NA soil  
44  
45 points, however, were outside this correlation, indicating (1) probable differences between WEOC in S-NA and  
46  
47 WEOC in SS-amended and/or TWW-irrigated soils, (2) that the increase in SOM due to SS amendment does not  
48  
49 increase the SOM mobility. The S-WR soil, despite high SOM contents, showed low WEOC values, confirming  
50  
51 that the application of manure does not necessarily increase the soil WEOC (Angers et al. 2006).  
52  
53

54 SUVA<sub>254</sub> index, positively related to humification, and the E2/E3 index, positively related to low-  
55  
56 molecular-weight compounds, were 6.9 and 8.5, respectively, for the TWW and 0.8 and 6.0, respectively, for the  
57  
58 SS water extract. The SUVA<sub>254</sub> value was high in TWW with regard to usually observed (Michael-Kordatou et  
59  
60 al. 2015) but in the usual range for the SS water extract (Xing et al. 2012). These values of SUVA and E2/E3,  
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253 however, can not be expected to have a significant impact on SS-amended and/or TWW-irrigated soils because  
1  
2 254 of the high biological activity in the topsoil modifying the SOM after SS amendment or TWW irrigation. In the  
3  
4 255 soil WEOC the SUVA and E2/E3 were in the usual range (Guigue et al. 2015). The SS-amended and/or TWW-  
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6 256 irrigated soils showed a decrease of SUVA with depth, as frequently observed (Hassouna et al. 2010), due to a  
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8 257 higher proportion of lignin derivative in the topsoil.

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### 11 12 13 259 **Water extractable nutrients**

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18 261 Nutrient concentration in TWW and in water extracts from SS and from the top 0-40 cm of the studied soils  
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20 262 are given on Fig. 3. The sewage sludge had high amounts of leachable  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$  and  $\text{SO}_4^{2-}$  and lower, but  
21  
22 263 non-negligible, concentrations for the sum of nitrogen species and  $\text{PO}_4^{3-}$ . The TWW brought also significant  
23  
24 264 concentrations of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{K}^+$ , but is poor in nitrogen. These characteristics are likely related to the high  
25  
26 265  $\text{K}^+$ ,  $\text{SO}_4^{2-}$  and  $\text{PO}_4^{3-}$  values observed for the S-SS, S-TW and S-STW soils when compared to the reference S-  
27  
28 266 WR and S-NA soils. The higher value of the  $\Sigma\text{N}$  (sum of nitrogen species) in the S-SS soil (0.2 % w/w) can also  
29  
30 267 be related to N input by SS (3.5 % w/w). The lower concentrations of  $\Sigma\text{N}$  in the water extracts of the S-TW and  
31  
32 268 S-STW soils with regard to the S-SS soil is more difficult to explain, it can be due to increased leaching of native  
33  
34 269 soil organic N because of an higher hydraulic loading from the effluent irrigation (Barton et al. 2005; Belaid et  
35  
36 270 al. 2012). Nitrogen species were mainly  $\text{NO}_2^-$  and  $\text{NH}_4^+$  in SS and TWW and mainly  $\text{NO}_3^{2-}$  in soils, indicating  
37  
38 271 rapid oxidation of N species in the soil, in accordance to other studies on organic-waste mineralization in soils  
39  
40 272 (Odlare et al. 2011). Variations with depth of the water extractable nutrients (Fig. 4) showed that the impact of  
41  
42 273 the amendments is higher in the surface horizons. In conclusion, it was proved that SS-amendment or TWW-  
43  
44 274 irrigation increased the nutrients disponibility, especially regarding K and P, in accordance with previous studies  
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46 275 with manure applied to soils (Laird 2008; Lehmann et al. 2003) or sludge from wastewater (He et al. 2001).

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### 50 51 277 **Bioavailability of trace elements**

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56 279 As sludge and/or TWW application resulted in a pH raise, the final pH being comprised between 7.2 and  
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58 280 7.7, there may be the question of the bioavailability of iron and of the trace-elements that are usually poorly  
59  
60 281 soluble in alkaline soils (Cu, Zn, Mn, Co, Ni) (Brallier et al. 1996; Kabata-Pendias and Pendias 2001). On the



282 other hand, the sludge brought around 155, 370, 19 and 200 mg kg<sup>-1</sup> for Cu, Zn, Co and Ni, respectively. The  
283 TWW brought around 14 µg L<sup>-1</sup> of Cu, the other trace elements being below the detection limit.

284 Results of water- and sodium-acetate extractions from soil material are given on Table 3, unfortunately Mn  
285 results were unreliable so that they are not given. The water-extraction gives the element concentration likely to  
286 be found in the soil solution after a rainfall, the sodium-acetate extraction gives an evaluation of the element  
287 bioavailability in the rhizosphere, were complexing compounds released by roots or associated micro-organisms  
288 can solubilize adsorbed or precipitated elements (Hinsiger 1998). Here Cu and Zn were more bioavailable in the  
289 SS-amended and/or TWW-irrigated soils than in the reference S-NA soil. Concerning Ni, however,  
290 concentrations in most extracts from the SS-amended and/or TWW-irrigated soils were below the detection  
291 limit, and below the values found for the reference S-NA soil.

292 Regarding iron, the amorphous solid phase is the more likely to be dissolved by complexing compounds in  
293 the rhizosphere, as usual low-weight carboxylic acids or microbial siderophore (Kraemer 2006). Here the  
294 amorphous iron phase extracted by a citrate/ascorbate solution corresponded to 1633, 373, 617 and 415 mg kg<sup>-1</sup>  
295 for the S-SS, S-TW, S-STW and S-NA soils, respectively. There was a slight diminution of the potentially  
296 bioavailable Fe in the soil that received TWW only, but a significative increase in soil that received sludge,  
297 likely because of the iron brought by the sludge where the Fe content is usually higher than 500 mg kg<sup>-1</sup> (Sahoun  
298 et al. 2012).

299

### 300 **Principal components analysis (PCA)**

301

302 Results of the PCA are given on Fig. 5. In this representation, positive correlation between variables is the  
303 higher when the variable representative points are close together and close to the circle, negative correlation the  
304 higher when points are opposite with regard to the circle center and close to the circle. When the variable  
305 representative points are orthogonal with regard to the circle center, they are uncorrelated. The two first factorial  
306 axes encompassed 66% of the total variance, indicating a satisfactory separation of the variables in the  
307 corresponding projection. The projection confirms and clarifies most of the conclusions given above. Water-  
308 extracted main nutrients (N, P, K, Mg) were correlated one to another and their weight on the first axis was  
309 driven by the SS-amended and/or TWW-irrigated soils, overall their upper horizons. There was no relationship  
310 between these parameters and SOC and CEC, that were more driven by the specificity of the S-WR and S-NA



311 soils. There was no correlation between water-extracted main nutrient and water-extracted Na and Cl,  
1 312 confirming the sufficient leaching of salt in SS-amended and/or TWW-irrigated soils. The negative correlation  
2 313 between water-extracted main nutrients and density confirmed the negligible effect of the inputs on soil  
3 314 compaction.  
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8 316

## 11 316 **Conclusion**

13 317

16 318 On Mediterranean areas that have been submitted for more than 10 years to irregular and relatively poorly  
17 319 controlled TWW irrigation and SS amendments, the agronomic quality of the soils has not decreased. Both SS  
18 320 amendment and TWW irrigation increased the nutrients amounts and availability, without altering other soil  
19 321 quality parameters such as CEC, salinity, *SAR*, *ESP*, SOM content or SOM C/N ratio. Soil permeability and trace  
20 322 elements bioavailability remained also satisfactory. SOM and total N were higher in soils that received SS only,  
21 323 likely because no priming effect or leaching. These observations highlight the potential benefits of application of  
22 324 wastewater and sewage sludge for improving the agronomical properties of the soils, even without quite  
23 325 restrictive procedures. From an environmental point of view, however, a potential contamination of soils with  
24 326 heavy metals needs to be evaluated; it will be the subject of a second part of this study.  
25 327

35 327

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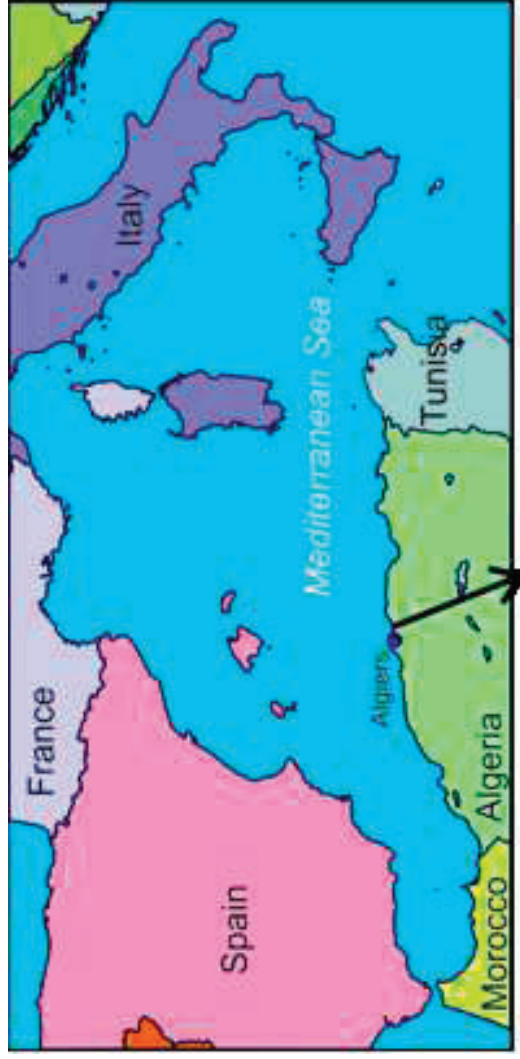


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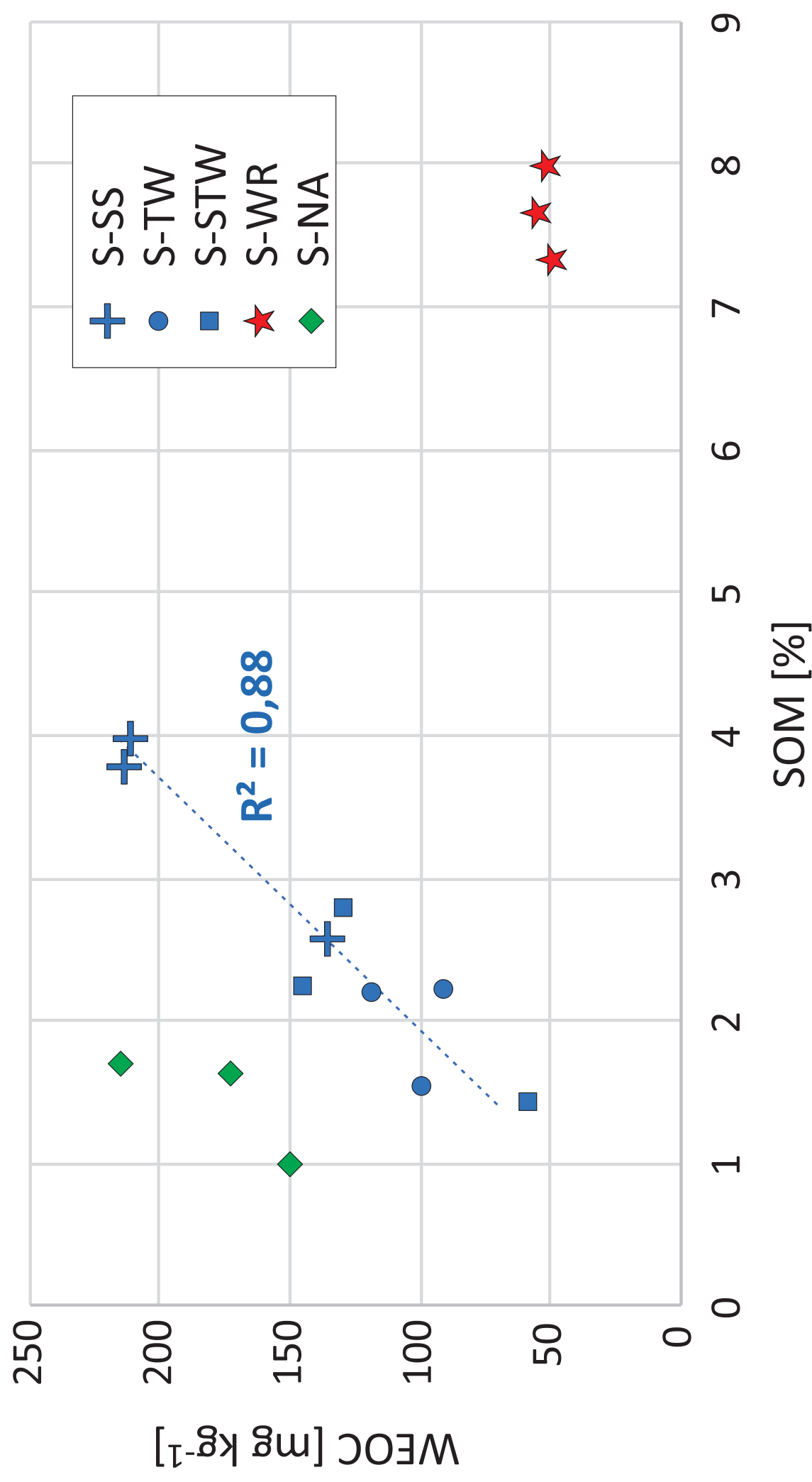


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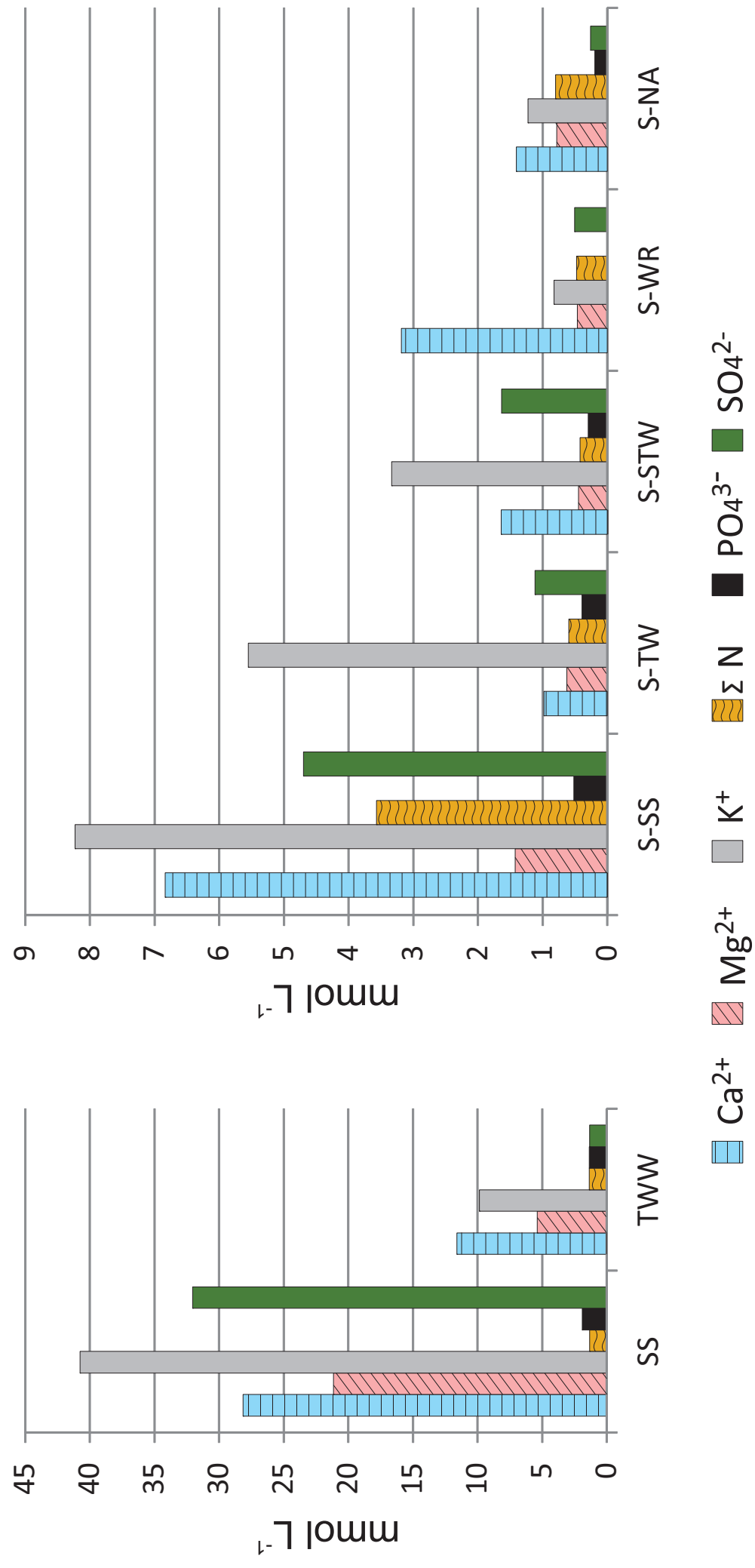




Figure

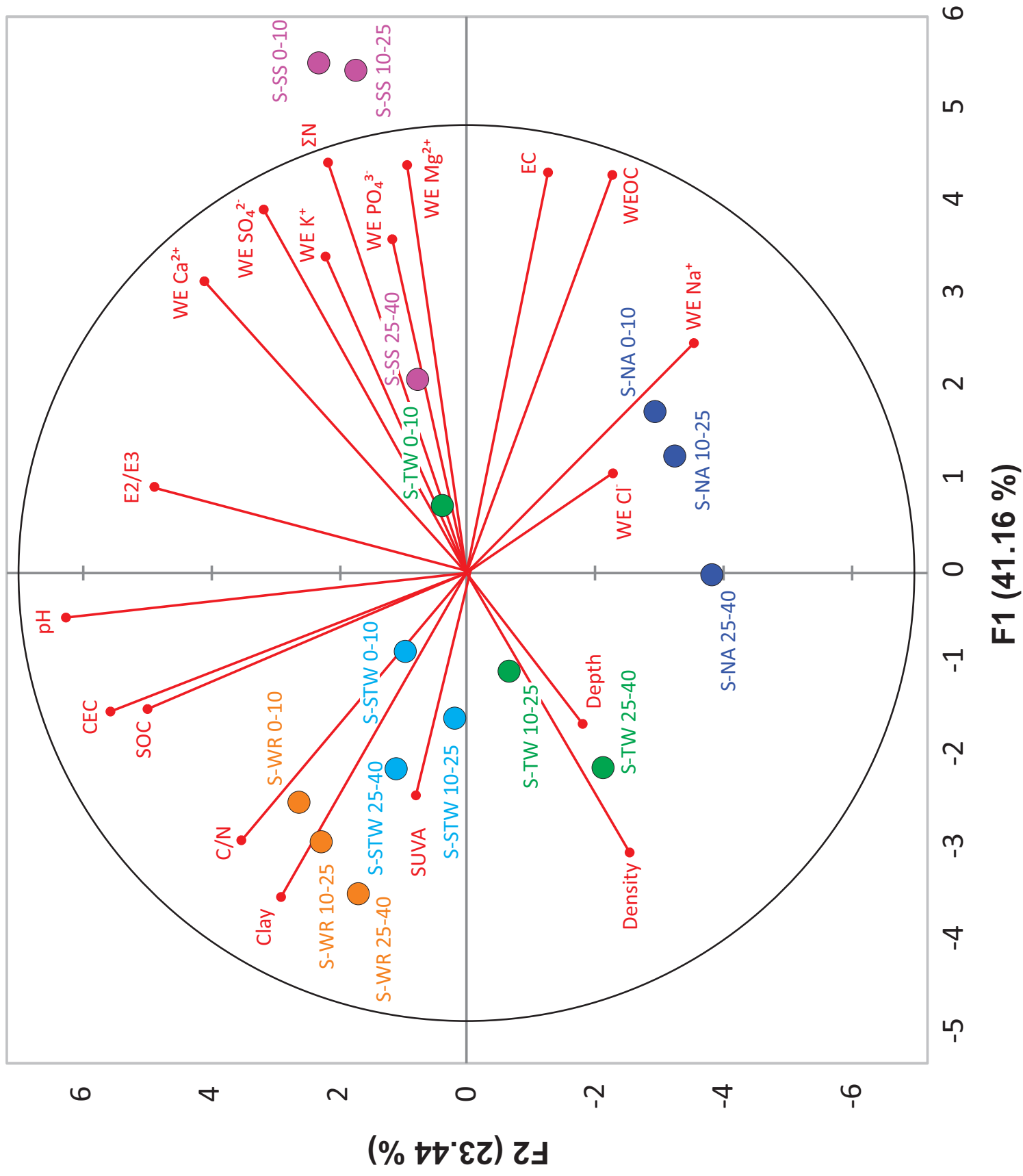


Figure



Figure

Biplot (F1 and F2 : 64.61 %)



Figure

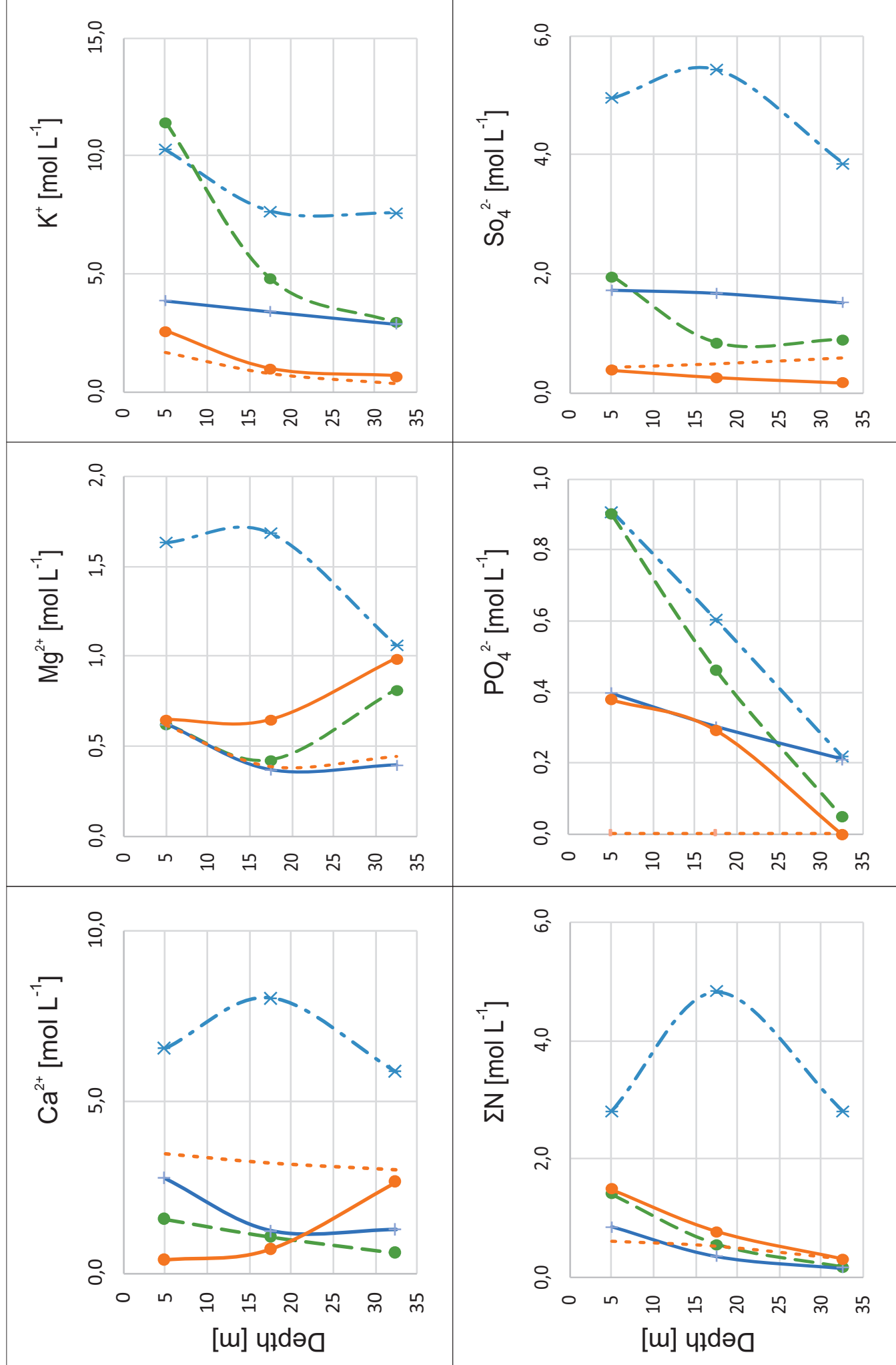


Table 1. Agronomic parameters for sludge, TWW and soils. Weighted average values for the 0-40 cm soil layer.

Parameter	Unit	S-SS	S-TW	S-STW	S-WR	S-NA	SS	TWW
Clay	%, w/w	17.4	21.6	36.8	30.9	12.7	#	#
Silt	%, w/w	44.9	51.3	30.1	42.8	55.8	#	#
Sand	%, w/w	37.7	27.2	33.1	26.3	31.5	#	#
$\rho_d$	$\text{g cm}^{-3}$	1.3	1.4	1.5	1.4	1.4	#	#
CEC	$\text{meq } 100\text{g}^{-1}$	15.8	11.4	19.4	19.6	9.8	38.2	#
SOC	%, w/w	2.0	1.1	1.2	4.4	0.8	22.5	#
N total	%, w/w	0.2	0.1	0.1	0.1	0.1	3.5	#
C/N		9.7	13.2	10.7	45.0	9.3	6.4	#
pH		7.5	7.2	7.6	7.7	7.0	8.1	7.8
EC	$\mu\text{S cm}^{-1}$	#	#	#	#	#	#	296
EC <sub>1:10</sub>	$\mu\text{S cm}^{-1}$	67	27	29	33	55	677	#
EC <sub>SP</sub>	$\mu\text{S cm}^{-1}$	1585	643	693	769	1296	16014	#
SAR	$(\text{mmolc L}^{-1})^{0.5}$	1.4	1.2	2.2	1.2	3.8	4.5	15.2
ESP		2.5	1.3	1.6	1.2	5.8	8.4	#
Dissolved species in water extract for soils and SS and in TWW								
WEOC	$\text{mg kg}^{-1}$	181.8	104.8	110.5	52.0	179.4	3826.0	3.8
Ca <sup>2+</sup>	$\text{mg kg}^{-1}$	274.1	39.4	65.8	127.6	56.4	1128.6	465.2
Mg <sup>2+</sup>	$\text{mg kg}^{-1}$	34.6	15.2	10.9	11.3	19.0	513.8	130.8
K <sup>+</sup>	$\text{mg kg}^{-1}$	321.8	217.0	130.5	32.2	47.9	1593.1	385.2
Na <sup>+</sup>	$\text{mg kg}^{-1}$	90.2	34.5	72.8	53.1	130.9	733.7	1439.9
Cl <sup>-</sup>	$\text{mg kg}^{-1}$	150.4	114.6	99.5	141.2	208.0	967.5	2061.7
F <sup>-</sup>	$\text{mg kg}^{-1}$	3.8	1.6	8.1	5.3	11.6	8.3	2.0
NO <sub>3</sub> <sup>-</sup>	$\text{mg kg}^{-1}$	179.7	28.3	16.7	22.4	26.3	14.6	1.9
NO <sub>2</sub> <sup>-</sup>	$\text{mg kg}^{-1}$	4.8	1.4	4.8	2.3	1.8	25.4	0.0
NH <sub>4</sub> <sup>+</sup>	$\text{mg kg}^{-1}$	1.3	0.6	<dl	<dl	4.7	9.2	24.2
PO <sub>4</sub> <sup>3-</sup>	$\text{mg kg}^{-1}$	49.2	37.5	28.0	0.0	18.6	181.8	130.1
SO <sub>4</sub> <sup>2-</sup>	$\text{mg kg}^{-1}$	450.9	107.1	156.7	48.4	24.7	3075.9	126.6





Table 2. Variation with depth of soil main characteristics.

Depth [cm]	SOM [% w/w]	WEOC in SOC			EC [ $\mu\text{S cm}^{-1}$ ]	pH	CEC [ $\text{cmol}_c \text{k}^{-1}$ ]	Clay	Silt	Sand	$\rho_d$ [ $\text{g cm}^{-3}$ ]
		% of SOC	SUVA <sub>254</sub>	E2/E3							
<b>S-SS</b>											
0-10	4.0	0.91	3.9	6.1	72.0	7.7	16.7	15.6	52.6	31.9	1.1
10-25	3.8	0.97	3.7	6.4	71.8	7.5	16.4	13.5	48.6	37.7	1.3
25-40	2.6	0.90	3.6	6.3	58.8	7.5	14.5	22.5	36.0	41.5	1.3
<b>S-TW</b>											
0-10	2.2	0.71	4.6	5.9	38.7	7.3	11.8	18.6	52.8	28.5	1.3
10-25	2.2	0.93	2.9	3.1	25.6	7.3	12.3	19.3	53.3	26.9	1.4
25-40	1.6	1.11	2.2	2.9	21.0	7.0	10.4	25.6	47.8	26.4	1.5
<b>S-STW</b>											
0-10	2.8	0.80	7.2	5.1	38.9	7.6	19.9	40.0	25.7	34.3	1.3
10-25	2.2	1.12	4.7	4.2	26.4	7.5	19.7	36.7	28.3	35.1	1.6
25-40	1.4	0.7	5.1	9.0	25.8	7.6	18.6	34.7	35.0	30.3	1.5
<b>S-WR</b>											
0-10	8.0	0.11	6.6	6.8	34.4	7.8	19.4	31.0	46.0	23.0	1.3
10-25	7.7	0.12	6.6	3.2	32.0	7.8	19.7	30.0	36.0	34.0	1.3
25-40	7.3	0.12	7.5	3.6	31.8	7.5	19.5	31.0	46.0	20.0	1.4
<b>S-NA</b>											
0-10	1.6	1.83	3.7	2.4	78.2	7.1	9.6	7.3	55.5	37.2	1.3
10-25	1.7	1.7	5.1	2.3	65.6	7.0	10.4	7.7	60.7	34.4	1.4
25-40	1.0	1.0	8.3	2.5	54.8	6.9	9.3	21.6	52.8	25.6	1.5
<b>SS</b>											
			0.8	6							
<b>TWW</b>											
			6.9	8.5							



Table 3. Trace element extraction, average in the 0-40 horizons, values in mg kg<sup>-1</sup>. W-ex.: water-extractable; Na-ac-ex.: sodium acetate extractable.

		S-SS	S-TW	S-STW	S-WR	S-NA
Cu	W-ex	0,39	1,03	0,76	0,14	0,41
	Na-ac-ex	2,03	10,86	4,90	0,28	0,74
Zn	W-ex	0,02	0,07	0,18	0,16	0,16
	Na-ac-ex	5,94	0,74	1,39	0,29	0,60
Co	W-ex	<dl	0,03	0,04	0,11	0,00
	Na-ac-ex	0,14	0,08	0,12	0,12	0,00
Ni	W-ex	<dl	<dl	0,03	0,07	0,06
	Na-ac-ex	<dl	<dl	<dl	<dl	0,11



## Figure captions

Fig. 1. Location of the sampling sites in the Boumerdes region, Northern Algeria. The point in the view is N 36°45'34.56", E 3°25'24.96". View from Google Earth, 2017.

Fig. 2. Water extractible organic carbon versus soil organic matter. The regression line concerns the S-SS, S-TW and S-STW points.

Fig. 3. Nutrients in TWW and in the water extract of SS and of the studied soils (average for the 0-40 cm topsoil).

Fig. 4. Variation with depth of water extracted nutrients.

Fig. 5. PCA correlation circle, variables (points) and samples (circles). Percent on each factorial axis gives the explained variance. WE: water extractible.

