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Sewage sludge use in bioenergy production. A case study of its effects on soil properties under *Cynara cardunculus* L. cultivation

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Abstract

Energy crops cultivation is expected to further increase, which represents an opportunity to establish synergies able to enhance key environmental components (*i.e.* soil). To reach this benefits crop management is crucial and should be properly assessed. The aim of this work is to provide an insight on the effects of sewage sludge compost (SSC) on soil properties, when this material is applied as basal dressing for the cultivation of a Mediterranean energy crop (*Cynara cardunculus* L.). A 3-years trial (2008/2011) was conducted in Alicante (Southeastern Spain), testing four SSC application rates (0, 30, 50 and 70 t/ha) on a heavy textured Anthrosol. The addition of SSC enhanced soil fertility, primarily increasing organic carbon (C_{ox}), Kjeldahl nitrogen (N_k), available P ($P_{Burriel}$), Cu_{DTPA} and Zn_{DTPA} levels. Comparatively with the control (0 t/ha), 30, 50 and 70 t/ha treatments induced a rise of 11%, 19% and 25% in N_k (Control=1.11 g/kg) and P_{Burriel} (Control=79 mg/kg), while for C_{ox} (Control=11.8 g/kg) was 14%, 21% and 30%. However, these variables apparently did not significantly decrease throughout the experiment, which suggests that the organic matter added was under a stabilization process, favoured by the poor physical properties of the soil. Other elements (Na_{NH4Ac}, K_{NH4Ac}, Mn_{DTPA}) were accumulated within the soil as time passed by, as a result of soil status, Mediterranean environmental conditions and crop management. The use of SSC as organic fertilizer represents an effective option to optimize cynara cultivation systems while improving soil quality through enhanced long-lasting organic matter pools.

Additional key words: sewage sludge compost; sustainability; cardoon; wastewater treatment by-products; organic amendment; energy crop management; soil protection.

Abbreviations used: $CaCO_{3eq}$ (equivalent calcium carbonate); C_{ox} (oxidable organic carbon); EC (electrical conductivity); EU (European Union); IVIA (Valencian Institute of Agricultural Research); N_k (Kjeldahl nitrogen); P_{Burriel} (available P for the plant extracted with the Burriel-Hernando method); SOM (soil organic matter); SS (sewage sludge); SSC (sewage sludge compost); X_{DTPA} (element extracted with diethylenetriaminepentaacetic acid, DTPA); X_{NH4Ac} (element extracted with ammonium acetate, NH₄Ac). **Citation:** Lag-Brotons, A. J.; Gómez, I.; Navarro-Pedreño, J. (2015). Sewage sludge use in bioenergy production. A case study of its effects on soil properties under *Cynara cardunculus* L. cultivation. Spanish Journal of Agricultural Research, Volume 13, Issue 1, e11-001, 14 pages. http://dx.doi.org/10.5424/sjar/2015131-6145.

Received: 24 Apr 2014. **Accepted:** 16 Jan 2015.

http://dx.doi.org/10.5424/sjar/2015131-6145

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Funding: This research was supported by the Spanish Ministry of Innovation and Science through a research fellowship (AP2007-01641) and by Miguel Hernández University as hosting institution.

Competing interests: The authors have declared that no competing interests exist.

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Introduction

Energy crops cultivation can potentially improve key ecosystem components, such as the soil, through synergies implemented by sustainable management criteria. Concerns about energy security and environmental threats (*i.e.* CO_2 effect on climate change) have led to strategies oriented towards sustainability, as the Directive 2009/08/EC, which established legally binding targets within the European Union (EU) for 2020 in order to reach a 20% share of renewable energy on the final energy consumption. Within the renewable energy pool, energy obtained from biomass is expected to increase considerably, being partially fulfilled by dedicated energy crops grown in abandoned or marginal lands (Bentsen & Felby, 2012). Energy crops cultivation aims to maximize biomass feedstock obtained per unit of area, minimize production inputs, and avoid land competition with edible crops. Complementary to these traits, several environmental cobenefits can be achieved through energy crops cultivation, such as the protection of soil, the increase in the terrestrial carbon sinks and reservoirs and the reduction of greenhouse gases emissions (Sims *et al.*, 2006). In order to achieve these environmental benefits key ecosystem components, as the soil, should be considered within energy crops management. Soil quality is regarded as an essential indicator of the sustainability of the agro-systems (Muller, 2009) and is related with the quality and the quantity of soil organic matter (SOM) (Karlen et al., 2001). In Mediterranean regions, SOM stocks are low or very low (Albaladejo et al., 2013), thus being prone to soil degradation (Loveland & Webb, 2003) and, consequently, vulnerable versus desertification processes. Moreover, it is expected that climate change will aggravate the conditions of Mediterranean environments, mainly due to an increase in the frequency of extreme events (*i.e.* droughts) and due to the rise of temperature (IPCC, 2007), which will likely induce a decrease of SOM content. Therefore, the enhancement of SOM levels is advisable, especially in Mediterranean countries, and has been recognized as an efficient option to tackle soil degradation (Gobin et al., 2011). The implementation of this option within the context of energy crops cultivation turns out in the application of organic amendments to the soil, which partially fulfils crop nutritional requirements and improves soil quality.

Composted sewage sludge is a suitable material to be applied as an organic amendment to energy crops systems due to its beneficial effects in the soil-plant system. Sewage sludge (SS) is an organic by-product derived from the treatment of urban wastewater, whose production has increased considerably in the recent years as a result of higher population and stringent water quality standards (Fytili & Zabaniotou, 2008). Whilst its composition is dependent on wastewater treatment processes, in general terms, is characterized by high levels of organic matter, organic nitrogen, P, Fe and Zn (Fytili & Zabaniotou, 2008). Precisely for these characteristics, when SS is applied as an amendment, it enhances soil fertility and SOM levels, as well as potentially contributes to the short-term carbon sequestration (Soriano-Disla et al., 2010). Consequently, the preferred end use of SS in the EU is found in the agricultural sector, applying to the lands more than 30% of the total production (>10 \cdot 10⁶ t on dry basis) (Mahmoud et al., 2012). Regarding energy crops trials, it has been successfully applied as organic fertilizer, enhancing the quantity and the quality of the biomass produced (Mahmoud et al., 2012; Mañas et al., 2013), thus appearing as an interesting organic fertilizer to complement or even replace the inorganic fertilization usually applied to energy crops (Quaye & Volk, 2013). However, there are some drawbacks to take into consideration on SS land application, as the presence of heavy metals (Smith, 2009) and toxic elements for the plant (Noble & Roberts, 2004). An efficient option to minimize these undesirable effects is composting,

which reduces heavy metal availability and diminish the presence of plant pathogens and organic toxicants (Barker & Bryson, 2002; Noble & Roberts, 2004). As observed for SS, when sewage sludge compost (SSC) is applied to the soil, fertility and plant growth are enhanced (Casado-Vela et al., 2006; Larchevêque et al., 2006; Song & Ju Lee, 2010). However, SS and SSC differ significantly in a crucial trait: the nature of the organic matter. Uncomposted SS presents greater proportion of labile organic matter (Blagodatskaya & Kuzyakov, 2008) while in its composted form has greater proportion of humic-like substances which are resistant to biodegradation, thus remaining for a comparatively longer period within the soil (Pérez Lomas et al., 2010), slowly releasing nutrients as mineralization occurs (Gil et al., 2011). It should be considered that land application of SSC is particularly effective in Mediterranean areas due to the improvement of soil physical properties (García-Orenes et al., 2005) and SOM levels for a comparatively longer period, which contributes to cope with soil salinization and erosion (Tejada et al., 2006). Irrespective of SS form, the use of these waste materials results in inputs costs savings when compared with exclusively inorganic-based fertilization (Song & Ju Lee, 2010). Thereby, if the aim pursued with the use of organic residues is the improvement of the soil-plant system in a sustained and sustainable way, SSC appears to be more appropriate than SS applications for Mediterranean environments.

Agro-system dedicated to the production of bioenergy is an old concept that has gained importance in the recent decade. Just to mention the example of the species used in this experiment, the research on Cynara cardunculus L. (cynara) as a Mediterranean energy crop for the production of biomass started in the 1980's (Fernández et al., 2006). Nowadays, the bibliography available is extensive, covering the production, the quality and the thermal behavior of cynara biomass (Monti et al., 2008; Angelini et al., 2009), as well as many other aspects of its industrial applications (Fernández et al., 2006). However, the effects of organic fertilization in cynara's cultivation systems are still uncertain. Inorganic fertilization has been tested, mostly based on N-fertilization treatments (Grammelis et al., 2008), even though K-fertilization treatments (Solano et al., 2010) and N-P-K fertilization treatments (Ierna et al., 2012; Mauromicale et al., 2014) have been also described. Concerning organic fertilization, Mañas et al. (2013) evaluated the effects of SS, while Lag-Brotons et al. (2014a) tested the effects of SSC, being both studies limited to the first year of cynara cultivation. Additionally, Lag-Brotons et al. (2014b) reported the effects of cynara's productivity in a three-year trial. From the aforementioned studies, just Lag-Brotons

et al. (2014a) reported soil data on the effects of fertilization treatments. Data describing the effects of cynara's fertilization treatments on soil properties beyond the first year of cultivation are needed in order to allow growers to evaluate the suitability of SSC use as organic amendment.

Motivated by the previous considerations, the main aims of this study were: i) to ascertain the effects of SSC on soil properties and; ii) the determination of the optimum SSC application dose while considering crop management effects on soil status.

Material and methods

Site description

A 3-year field experiment (2008-2011) was conducted in Alicante (38° 13' N, 0° 42', 98 m a.s.l.), South-East Spain. The soil was a heavy textured Anthrosol (Table 1), which had been used for the production of artichoke (*Cynara cardunculus* L. var. *scolymus*) and ornamental flowers. The local climate of the region is semiarid-Mediterranean with mild winters and hot rainless summers. On the 1999-2013 series (long-term), recorded near the experimental field (38° 14' N, 0° 41', 98 m a.s.l.) at an agro-meteorological station of the Valencian Institute of Agricultural Research (IVIA), mean annual temperature and accumulated precipitation were 12°C and 262 mm, respectively. Rainfall, daily

Table 1	. Soi	and	sewage	sludge	compost	(SCC)	physico-
chemica	ıl proj	pertie	S				

Soil		Units	SSC		Units	
Clay	38	%	pH _{1:5}	6.8		
Silt	39	%	EC _{1:5}	6.4	dS/m	
Sand	23	%	Moisture	21	%	
CaCO _{3eq}	53	%	Cox	292	g/kg	
Active lime	194	‰	N_k	24	g/kg	
Bulk density	1.3	g/cm ³	P _{total}	706	mg/kg	
pH _{1:2.5}	8.6		Ca _{total}	19	g/kg	
EC _{1:5}	0.35	dS/m	K _{total}	4.8	g/kg	
C _{ox}	11.1	g/kg	Mg _{total}	5.8	g/kg	
N _k	1.1	g/kg	Na _{total}	2.6	g/kg	
P _{Burriel}	97	mg/kg	B _{total}	0.05	g/kg	
Ca _{NH4Ac}	2.86	g/kg	Cu _{total}	159	mg/kg	
Mg _{NH4Ac}	0.58	g/kg	Fe _{total}	5724	mg/kg	
K _{NH4Ac}	0.67	g/kg	Mn _{total}	129	mg/kg	
Na _{NH4Ac}	0.20	g/kg	Zn _{total}	690	mg/kg	
Cu _{DTPA}	2.4	mg/kg	Cd _{total}	0.8	mg/kg	
Fe _{DTPA}	4.0	mg/kg	Cr _{total}	60	mg/kg	
Mn _{DTPA}	2.3	mg/kg	Ni _{total}	13	mg/kg	
Zn _{DTPA}	2.7	mg/kg	Pb _{total}	47	mg/kg	
Cd _{total}	0.36	mg/kg				
Cr _{total}	23.0	mg/kg				
Ni _{total}	12.5	mg/kg				
Pb _{total}	11.7	mg/kg				

 $CaCO_{3eq}$: equivalent calcium carbonate; EC: electrical conductivity; C_{ox} : oxidable organic carbon; N_k : Kjeldahl nitrogen; NH_4Ac : element extracted with ammonium acetate; DTPA: element extracted with diethylenetriaminepentaacetic acid.

maximum and minimum air temperature during the experiment were recorded at the previously mentioned IVIA station and are shown in Fig. 1.



Figure 1. Rainfall and air temperature during the experimental period (2008-2011) and the mean value for the period 1999-2012 (long-term).

Experimental materials

The SSC was obtained from the municipal wastewater treatment plant of Aspe, Alicante (Spain). The compost consisted of SS mixed with sawdust and straw as co-composting agents in an approximate proportion of 4:3:1 (v/v). Compost samples were analyzed under the recommended standards in Spanish Royal Decree 824/2005, in its annex VI (BOE, 2005). The characteristics of SSC are shown in Table 1. Irrigation water consisted in a mixture of good quality water (interbasin transfer water of Tajo-Segura) and medium quality water (secondary treatment water from Algoros wastewater treatment plant) and presented the following characteristics: EC, 1.8 dS/m; pH, 7.9; N_k, <5 mg/L; chemical oxygen demand, 34 mg O₂/L; biochemical oxygen demand₅, 9 mg O₂/L; Cl⁻, 301 mg/L; NO₃⁻, 7 mg/L; HCO₃⁻, 177 mg/L; NH₄⁺, 8.1 mg/L; B, 0.7 mg/L; Ca²⁺, 53 mg/L; K⁺, 17 mg/L; Mg⁺², 49 mg/L; Na⁺, 190 mg/L. Cynara seeds (*Cynara cardunculus* L. var. altilis DC - cultivated cardoon) were commercially obtained.

Experimental set-up

In a completely randomized block experimental design with three replications at similar locations, four SSC application rates were assessed. Each plot was split into four subplots (9.6 m^2 each), corresponding to the different SSC treatments. Compost was applied as an organic amendment that substituted the inorganic basal dressing normally done before plant establishment. The SSC treatments were designed to enhance soil organic carbon, avoid excessive N-fertilization and not surpass heavy metals application limits of the Spanish Royal Decree 1310/1990 (BOE, 1990), which for soils with pH>7 are (mg/kg of dry matter): Cd=3; Cu=210; Ni=112; Pb=300; Zn-450; Cr=150. The following application rates, expressed in t SSC/ha, were tested: 0 (T₀), 30 (T₁), 50 (T₂) and 70 (T₃). It was assumed that 15% of SSC organic N-content would be released (Gil et al., 2011; López-López et al., 2012). Consequently, the corresponding N-fertilization rates of SSC treatments were 0, 85, 142 and 200 kg-N/m². Compost treatments were similar to the typical SSC dosage applied on field experiments (Casado-Vela et al., 2006; Larchevêque et al., 2006; De Andrés et al., 2007). The incorporation of SSC into the soil was done by ploughing to a depth of 30 cm two weeks prior transplanting (year 2008/2009). Twelve cynara seedlings per SSC treatment were transplanted into the field on October 31 2008 (48 seedlings per plot), in a 1.0×0.8 m format (12,000 plants/ha). Irrigation was carried out between November

and June, maintaining the total amount of water available for the plant (rainfall + irrigation) at 760 mm/year, approximately. Every year 50-80-100 kg/ha of $N-P_2O_5-K_2O$ were applied along with irrigation water in order to restore nutrients extracted by the crop.

Data collection

Soil samplings were carried out every four months, starting two weeks after the incorporation of SSC to the soil and finishing on July 2011 (9 samplings). From each subplot, four soil samples were randomly taken (16 samples per plot) after removing vegetable material from the soil surface. Soil samples were collected up to 15 cm depth, air-dried at room temperature and sieved to pass through a 2 mm mesh. Then, they were stored in polyethylene bags and maintained at constant temperature ($\approx 15^{\circ}$ C) until further analysis. Soil pH and EC determinations were carried out in soil/deionised water suspension of 1:2.5 and 1:5 (w/v) respectively (MAPA, 1986). Oxidable organic carbon (C_{ox}) was determined by the Walkey Black method (Nelson & Sommer, 1996) while nitrogen contained in the organic fraction of soil (N_k) was analyzed by the Kjeldahl method (Bremner, 1965). Available phosphorous (P_{Burriel}) was determined using the Burriel-Hernando method (Díez, 1982). Concerning the available elements for the plant, Ca, K, Mg, and Na were extracted with ammonium acetate extract (NH_{4Ac}) while micronutrients (Cu, Fe, Mn and Zn) were extracted with diethylenetriaminepentaacetic acid (DTPA) (Lindsay & Norvell, 1978). In these soil extracts, Ca_{NH4Ac} , Mg_{NH4Ac} and DTPA-extracted micronutrients were measured by ion absorption spectrometry, while Na_{NH4Ac} and K_{NH4Ac} were measured by ion emission spectrometry.

Data analysis

The main aim of data analysis was to ascertain the effects of SSC applications on soil properties (SSC effects). In addition, the evolution of soil variables throughout the experimental period (time effects) was considered. Studied factors (SSC, time and their interaction) were assessed by using a two-way Analysis of Variance (ANOVA) (p<0.05). Whenever a variable was demeed as significant, means were separated by using Duncan's range test (p<0.05). In addition, Pearson correlations (p<0.05) were calculated between soil variables in order to further explain the effects observed throughout time. Soil data were log10-transformed prior to ANOVA and Pearson's correlation analysis in order to meet normality. Mean values, standard devia-

tions and standard errors of untransformed data are reported. Statistical analyses were calculated by using SPSS software (v 21.0; Armonk, NY; IBM Corp).

Results

Significant differences (p<0.05) due to the effect of SSC treatments and time were observed, while the interaction of these factors resulted non-significant (Table 2). To a lesser or greater extent, the totality of soil variables varied with time. Similarly, SSC treatments affected most of studied variables, with the exception of Ca_{NH4Ac}, K_{NH4Ac} and Na_{NH4Ac} (Table 2).

Concerning C_{ox} , N_k and $P_{Burriel}$ concentration, both SSC treatments influenced the levels of these elements (Table 2). However, while the increasing trend due to SSC treatments was fairly observable, especially for C_{ox} and N_k (Table 3, Fig. 2), the trend due to the pass of time was not so evident (Fig. 2). In fact, it was observed a scarce increase when the C_{ox} and N_k levels at 24-28 months were compared with the previous soil samplings (data not shown). Whilst significant, the deviations from the mean (Fig. 2) suggested that it was a minor effect, thus being SSC treatments the primary effect. The increment observed in the concentration of N_k and $P_{Burriel}$ regarding control value (T_0) of T_1 , T_2 , and T_3 soil samples was 11%, 19% and 25%, respectively, while that of C_{ox} was 14%, 21% and 30%, respectively.

Regarding ammonium acetate–extracted elements (Ca_{NH4Ac} , Mg_{NH4Ac} , K_{NH4Ac} and Na_{NH4Ac}), the influence of time was determining while SSC treatments effects were marginal, except for Mg_{NH4Ac} (Table 2). Whilst Mg_{NH4Ac} concentration was slightly affected by SSC treatments, Ca_{NH4Ac} , K_{NH4Ac} and Na_{NH4Ac} were not affected by the application of composted materials (Tables 2 and 4). Concerning time effects, none of these elements showed a clear trend, yet the concentration of Na_{NH4Ac} and K_{NH4Ac} , comparatively with the beginning of the experiment, apparently tend to increase (Fig. 3). The concentration of K_{NH4Ac} started at 0.7 g/kg (24 months), then increased up to a maximum of 1.7 g/kg (24 months), to finally end at a concentration of Na_{NH4Ac} remained stable (approxi-

 Table 2. Two-way ANOVA F-test statistics for differences caused by sewage sludge compost (SSC) and time on soil properties

Variable	SSC effects	Time effects	SSC × Time
pH _{1:2.5}	F _{3,396} =32.69; <i>p</i> <0.001	F _{8,396} =50.11; p<0.001	$F_{24,396} = 1.273; p=0.177$
$EC_{1:5}$	F _{3,396} =10.56; <i>p</i> <0.001	F _{8,396} =57.38; <i>p</i> <0.001	$F_{24,396} = 1.307; p=0.154$
C _{ox}	F _{3,396} =142.0; <i>p</i> <0.001	$F_{8,396} = 11.15; p < 0.001$	$F_{24,396} = 0.888; p=0.620$
N _k	F _{3,396} =71.55; <i>p</i> <0.001	$F_{8,396} = 8.72; p < 0.001$	$F_{24,396} = 0.703; p=0.849$
P _{Burriel}	F _{3,396} =16.14; <i>p</i> <0.001	$F_{8,396} = 6.22; p < 0.001$	$F_{24,396} = 0.256; p=1.000$
Ca _{NH4Ac}	F _{3,396} =0.17; <i>p</i> =0.917	$F_{8,396} = 144.4; p < 0.001$	$F_{24,396} = 0.120; p=1.000$
Mg _{NH4Ac}	F _{3,396} =6.62; <i>p</i> <0.001	$F_{8,396} = 45.97; p < 0.001$	$F_{24,396} = 0.695; p=0.857$
K _{NH4Ac}	F _{3,396} =0.56; <i>p</i> =0.640	F _{8,396} =82.63; <i>p</i> <0.001	F _{24,396} =0.428; <i>p</i> =0.993
Na _{NH4Ac}	F _{3,396} =1.75; <i>p</i> =0.157	$F_{8,396} = 224.5; p < 0.001$	F _{24,396} =0.539 ; <i>p</i> =0.965
Cu _{dtpa}	F _{3,396} =19.75; <i>p</i> <0.001	$F_{8,396} = 20.05; p < 0.001$	$F_{24,396} = 0.410; p=0.995$
Fe _{dtpa}	F _{3,396} =7.99; <i>p</i> <0.001	$F_{8,396} = 71.47; p < 0.001$	$F_{24,396} = 0.855; p=0.665$
Mn _{DTPA}	F _{3,396} =5.01; <i>p</i> <0.005	$F_{8,396} = 548.9; p < 0.001$	$F_{24,396} = 1.543; p=0.050$
Zn _{DTPA}	$F_{3,396} = 146.6; p < 0.001$	$F_{8,396} = 60.86; p < 0.001$	$F_{24,396} = 0.510; p=0.975$

EC: electrical conductivity; C_{ox} : oxidable organic carbon; N_k : Kjeldahl nitrogen; NH_4Ac : element extracted with ammonium acetate; DTPA: element extracted with diethylenetriaminepentaacetic acid.

Table 3. Physicochemical properties of studied soil as affected by sewage sludge compost (SSC) treatments. The mean value and the standard error (between brackets) throughout the 3-years of experiment are shown (n=108).

SSC	EC	рН	C _{ox}	N _k	P _{Burriel}
treatments	(dS/m)		(g/kg)	(g/kg)	(mg/kg)
$T_0 (0 t/ha)$	0.76 (0.05) a	8.35 (0.02) c	11.8 (0.1) a	1.11 (0.01) a	79 (2) a
$T_1 (30 t/ha)$	0.93 (0.07) bc	8.24 (0.02) b	13.4 (0.1) b	1.23 (0.01) b	88 (2) b
$T_2 (50 t/ha)$	0.98 (0.06) c	8.17 (0.02) a	14.3 (0.1) c	1.33 (0.02) c	94 (2) c
$T_3(70 \text{ t/ha})$	0.81 (0.05) ab	8.19 (0.02) a	15.3 (0.2) d	1.39 (0.02) d	98 (2) c

Different letters within a column indicate significant differences according to Duncan's range test (p<0.05). EC: electrical conductivity; C_{ox} : oxidable organic carbon; N_k Kjeldahl nitrogen; $P_{Burriel}$: available P extracted using Burriel-Hernando method.



Figure 2. Organic carbon (a), Kjeldahl nitrogen (b) and available phosphorous $(P_{Burriel})$ (c) soil concentration throughout the experimental period. Each bar colour indicates the sewage sludge compost treatment used. Mean values and standard deviation are shown (n=4).



0 t/ha 📕 30 t/ha 📕 50 t/ha 📕 70 t/ha Figure 3. Ammonium acetate extracted elements (Ca, K, Mg and Na) soil concentration throughout the experimental period. Each bar colour indicates the sewage sludge compost treatment used. Mean values and standard deviation are shown (n=4).

16

Months

20

0

4

32

Table 4. Concentration of the available elements extracted with ammonium acetate (Ca_{NH4Ac} , Mg_{NH4Ac} , K_{NH4Ac} and Na_{NH4Ac}) and diethylenetriaminepentaacetic acid (Cu_{DTPA} , Fe_{DTPA} , Mn_{DTPA} and Zn_{DTPA}) in the studied soil as affected by sewage sludge compost (SSC) treatments. The mean value and the standard error (between brackets) throughout the 3-years of experiment are shown (n=108)

SSC treatments	Ca _{NH4Ac} (g/kg)	K _{NH4Ac} (g/kg)	Mg _{NH4Ac} (g/kg)	Na _{NH4Ac} (g/kg)	Cu _{dtpa} (mg/kg)	Fe _{DTPA} (mg/kg)	Mn _{DTPA} (mg/kg)	Zn _{DTPA} (mg/kg)
T ₀ (0 t/ha)	2.83 (0.08) a	1.04 (0.04) a	0.57 (0.02) a	0.81 (0.06) a	3.0 (0.1) a	1.8 (0.1) a	9.8 (0.5) ab	3.2 (0.1) a
T ₁ (30 t/ha)	2.82 (0.08) a	1.09 (0.05) a	0.61 (0.02) b	0.85 (0.06) a	3.1 (0.1) ab	1.8 (0.1) a	9.7 (0.5) a	3.9 (0.1) b
$T_2(50 \text{ t/ha})$	2.87 (0.08) a	1.07 (0.05) a	0.63 (0.02) b	0.84 (0.06) a	3.3 (0.1) b	1.8 (0.1) a	10.1 (0.5) bc	4.4 (0.1) c
T ₃ (70 t/ha)	2.85 (0.08) a	1.02 (0.04) a	0.63 (0.02) b	0.82 (0.06) a	3.6 (0.1) c	2.1 (0.1) b	10.6 (0.5) c	4.7 (0.1) d

Different letters within a column indicate significant differences according to Duncan's range test (p < 0.05).

mately 0.2 g/kg) during the first year of cultivation (12 months). Then increased up to 1.1 g/kg and stayed at that value until the last sampling, when it increased up to 1.6 g/kg. In order to further explore the variations observed throughout time, the mean value irrespective of SSC treatment for each element within each soil sampling was calculated and represented in Fig. 4. As can be observed, Ca_{NH4Ac} was the element which, in general terms, contributed to a greater extent to these cations pool. In addition, Mg_{NH4Ac} decreased while K_{NH4Ac} and Na_{NH4Ac} increased their share to the general pool.

Micronutrients concentration increased as a result of SSC treatments (Table 4). The element that SSC treatments affected to a greater extent was Zn_{DTPA} (Table 2), whose concentration in T₃ was increased by 50% regarding control. The rest of the micronutrients (Cu_{DTPA}, Fe_{DTPA}, Mn_{DTPA}) were also affected, being the differences evident between T₀ and T₃ samples. Regarding time effects, Mn_{DTPA} and Zn_{DTPA} concentration increased, Fe_{DTPA} concentration decreased and Cu_{DTPA} concentration of Zn_{DTPA} increased as time passed by, especially from the



Figure 4. Variations on the concentration of Ca, K, Mg and Na extracted with ammonium acetate throughout the experimental period. Data shown are referred to the value for each soil sampling irrespective of the SSC treatments. For each element, the mean value and its corresponding standard deviation are shown (n=16).

sampling corresponding to 20 months onwards (Fig. 5). Comparatively with Zn_{DTPA} , the increase in the availability of Mn_{DTPA} due to time effects was far more evident (Table 2), as its concentration constantly increased from the start of the trial up to the 8th sampling (28 months) (Fig. 5). On average basis irrespective of SSC treatments, Mn_{DTPA} concentration started at 2.2 mg/kg and ended at 12.6 mg/kg. Concerning Fe_{DTPA}, it started at a concentration of 4 mg/kg, reaching a final concentration of 1.8 mg/kg (Fig. 5). It should be noted that the drastic reduction in Fe_{DTPA} availability from 0-4 months could be motivated by the abundance carbonates present in the studied soil (See Table 1).

The application of SSC treatments slightly decreased soil pH, yet did not clearly influence soil EC (Table 3). Generally, it could be expected that higher application rates of composted materials, thus enhanced salt loading, would lead to higher EC. However, this trend was not observed (See Table 3). Hence, if EC variations throughout time (Fig. 6) are considered, it could be assumed that time was the main driving factor regarding EC dynamics. This asseveration could be extended to pH dynamics also, as wider variations were observed for time rather than for SSC treatments. In order to ascertain the causes underlying EC variation and considering that EC values are related to the presence of salts in the soil solution, Pearson correlations between EC and the sum of cations (Ca, K, Mg and Na), as well as between EC and the sum of cations with greater solubility among those analyzed (K, Mg and Na) were calculated. The results indicated a stronger significant correlation between EC and the more soluble cations (n=432; r=0.697; p < 0.01) in comparison with the correlation between all the elements extracted with ammonium acetate and the EC values (n=432; r=0.573; p<0.01).

Discussion

The effects observed throughout the experimental period could be reasonably assumed to be related with crop management and with the Mediterranean traits of



Figure 5. Soil concentration of micronutrients extracted with DTPA (Cu, Fe, Mn and Zn) throughout the experimental period. Each bar colour indicates the sewage sludge compost treatment used. Mean values and standard deviation are shown (n=4).



Figure 6. Soil electrical conductivity and pH throughout the experimental period. Each bar colour indicates the sewage sludge compost treatment used (n=4).

the experimental plots soil. It was observed that soil EC and the concentration of Ca_{NH4Ac} , K_{NH4Ac} , Mg_{NH4Ac} , Na_{NH4Ac} and Mn_{DTPA} were mainly driven by time effects. In addition, the EC trend appeared to be linked with the variation in the concentration of K, Mg and Na. Generally, Mediterranean soils tend to present clayey texture, thus high water holding capacity, and low structural stability, which in many cases indicates low water infiltrability (Torrent, 2005). Considering that these features are likely to be present in the soil of the experimental plots (See Table 1), saturation conditions and impeded drainage might have occurred. If so, soil aeration would have been limited and soil conditions would have favored the reduction of Mn to the divalent

form, which is more soluble (El-Jaoual & Cox, 1998), leading to the sustained increase in Mn_{DTPA} concentration that was observed. Similarly, the probable reasons underlying the accumulation of Na_{NH4Ac} , a readily soluble element, close to soil surface were the combination of poor drainage and high evapotranspiration conditions, which are common factors of occurrence in arid and semi-arid regions (Sakadevan & Nguyen, 2010). In the present experiment, both Na and K, were primarily incorporated through irrigation water applied, being the accumulation of these elements a likely source that contributed to modify soil EC. Another fact indicative of the poor physical properties of the studied soil is the scarce variation observed in C_{ox} and N_k levels.

Considering that organic substrates added to the soil are biologically degraded (Vargas-García & Suárez-Estrella, 2008), those soil properties related to the organic fraction, such as Cox and Nk, are normally expected to decrease over time (Larchevêque et al., 2006), yet they remained fairly constant. The mineralization of SOM is dependent on the nature of the organic materials (Gil et al., 2011) but as well is strongly conditioned by environmental factors (Pérez Lomas et al., 2010). The climate conditions and soil characteristics of the experiment could have favoured organic matter stabilization (Pérez Lomas et al., 2010), by means of physical protection (decomposers impeded gas exchange and access to organic substrates) (Van Veen & Kuikman, 1990) and chemical protection (sorption and complexation interactions, enhanced by multivalents cations and clayey texture) (Jastrow et al., 2007). Hence, physicochemical protection can be contemplated as one of the main driving processes regarding C_{ox} and N_k dynamics in the present experiment. All the previous considerations could justify that time effects were mainly driven by soil quality (*i.e.* structure) according to the management (i.e. irrigation water quality) carried out.

The effects induced by SSC on soil properties were similar to those reported by several authors under Mediterranean conditions. Whilst limited to one year of duration, Lag-Brotons et al. (2014a) obtained practically the same results (decrease of pH and increase in C_{ox}, N_k, P_{Burriel}, Cu, Fe, Mn and Zn concentration) using a SSC of the same origin as that of this work. Similarly, in a 2.5 years field experiment, Lachervêque et al. (2006) reported an improvement of soil fertility (increase of SOM, N, P, Mg, K, Cu and Zn) as a result of SSC application, even though most of the effects declined to control level at the end of the study. Casado-Vela et al. (2006), additionally to the decrease of pH and the enhancement of soil fertility (SOM, N_k , P, Cu, Fe and Zn), reported an improvement of soil bulk density and microbial biomass in a 3-year trial as a consequence of the application of SSC to a semi-arid soil. The enhancement of soil fertility is caused directly by nutrients supplied by SSC and indirectly by the promotion of nutrient retention in the soil matrix (De Lucia *et al.*, 2013). In addition, the use of SSC as soil conditioner has been also reported to improve soil physical properties, both in non-salinized and salinized soils (García-Orenes et al., 2005; Tejada et al., 2006). As a result of the role of SSC as soil improver, the growth of a wide variety of crops has been enhanced (Casado-Vela et al., 2006; Larchevêque et al., 2006; De Andrés et al., 2007; Song & Ju Lee, 2010; De Lucia et al., 2013), among which cynara is included (Mañas et al., 2013; Lag-Brotons et al., 2014a). In fact, under the conditions

described in the present study, cynara's productivity was enhanced, improving biomass and seeds yield in approximately 40% and 68% (T_0 versus T_3 plants) (Lag-Brotons *et al.*, 2014b).

In the present work the beneficial effects of SSC applications were dependent on the rate used, being the optimum range comprised within 30 and 50 t SSC/ha. Considering that sewage sludge application, either treated (*i.e.* composted) or not, raises environmental concerns related to the presence of heavy metals (Smith, 2009), the amount should be optimized in order to apply the minimum quantity required to achieve beneficial effects. In this sense, certain soil properties (*i.e.* pH and P_{Burriel}) were not different in T₂ and T₃ samples, while most of them differed when T₁ and T₂ were compared. Therefore, in order to minimize the amount of heavy metals incorporated into the soil, an application dose of 50 t SSC/ha (T₂) was considered as preferable.

In Mediterranean agro-systems, the use of organic by-products, such as SSC, represents an economic feasible and environmentally desirable option for energy crops cultivation. Several studies have addressed the use of organic materials as amendments for energy crops cultivation (Mahmoud et al., 2012; Mañas et al., 2013; Quaye & Volk, 2013; Lag-Brotons et al., 2014a), indicating a growing interest towards the production of bioenergy under integrated management. Inorganic fertilizers are substantially responsible for the major part of carbon emissions, as well as account for a great share of the economic and energy costs (Dufour *et al.*, 2013). Therefore, the use of SSC could reduce the dependency on inorganic fertilizer while at the same time contribute to decrease greenhouse gases emissions (De Lucia et al., 2013). Additionally, SSC is a processed by-product of wastewater treatment, whose supply is guaranteed in the long-term and with potentially lesser cost (Song & Ju Lee, 2010). Furthermore, plants grown on SSC amended soils which are devoted to bioenergy production minimize toxicity risk to the food chain as are dedicated to non-edible uses. The aspect possibly of the outmost importance regarding SSC use in Mediterranean areas is the potential protection against common Mediterranean soil degradation factors by enhancing soil organic carbon. Aguilera et al. (2013) indicated that soil organic carbon is highly sensitive to changes in management under Mediterranean conditions, increasing its content under proper management practices (Aguilera et al., 2013). Within this last aspect, the selection of the crop species exerts an important role. As an example, cynara is a plant species which has been acknowledged as an effective protective agent against erosion, as it soon develops in autum, practically covering the whole soil surface for extended periods (Grammelis et al., 2008). In addition,

its perennial life cycle, which can last longer than 10 years (Angelini *et al.*, 2009), minimizes soil disturbation, as no tillage is applied during its cultivation, and favours the enhancement of SOM pool (Mauromicale *et al.*, 2014). Consequently, the cultivation of cynara encompassed by the application of stabilized organic matter sources, as SSC, is a management strategy which tends to maintain and preserve soil quality.

As final conclusions, the soil status was improved by the application of SSC as basal dressing in Cynara cardunculus L. cultivation for energy production. The optimum dose, according to effects induced on soil fertility, was comprised between 50 (T_2) and 70 (T_3) t SSC/ha, yet T_2 was considered preferable in order to minimize heavy metals loading. The concentration of the elements related to the organic fraction of soils (C_{ox} , N_k, P_{Burriel}) was enhanced, regarding control values, by compost amendments (T_1 -11%, T_2 -19% and T_3 -25% for N_k and $P_{Burriel}$, and T_1 -14%, T_2 -21% and T_3 -30% for C_{ox}), but, apparently, did not decrease as time passed by. This behaviour indicated that the organic matter added to the soil through SSC amendment was under stabilization rather than mineralization processes. The poor physical properties of the experimental soil probably favoured this dynamic, as well as contributed to the progressive rise of Na and Mn concentration. Typical effects of SSC amendment were also induced, such as micronutrients (Cu_{DTPA}, Fe_{DTPA}, Mn_{DTPA}, Zn_{DTPA}) increase and pH decreases. Thereby, the incorporation of SSC as basal dressing for cynara cultivation represents fertilization savings and a feasible option to effectively improve SOM levels, thus potentially enhancing soil quality and preventing its degradation.

Acknowledgements

Alfonso Jose Lag-Brotons gratefully acknowledges the Spanish Ministry of Innovation and Science for a research fellowship (AP2007-01641). The former author also acknowledges the collaboration and support of Algoros farmers, especially Pedro Valero. The authors also acknowledge the technical assistance of the Agrochemistry and Environment Department laboratory staff.

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