

# Effect of the addition of sewage sludge as a fertilizer on a sandy vineyard soil

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## Abstract

**Purpose** The application of sludge from wastewater in agriculture has increased in recent years, and it is therefore important to assess the effect that such treatment has on both the soil and the plant. The aim of the study described here was to ascertain whether there is a variation in the properties of the soil and to determine if this addition has an impact on the plant.

**Materials and methods** The area of investigation was close to the municipality of Villarrubia de los Ojos (Ciudad Real). In this work, six samples were taken from the surface horizon in the studied plot at a depth of 35 cm. A further three samples were taken: (i) a surface horizon of a soil close to the area under investigation but without treatment (control sample), (ii) a sample of sludge from the wastewater treatment plant and (iii) a sample of the mixture used by farmers as fertilizer. Laboratory tests were conducted in accordance with the SCS-USDA (1972) guidelines. Trace element samples were analysed by X-ray fluorescence spectrophotometry (Philips PW 2404).

**Results and discussion** The parcel of land studied is dominated by a sandy texture (88.3 % sand), and a decrease in pH was observed in areas in which the mixture (manure + sludge) was added (pH=8.0) compared to areas in which fertilizer was not

applied (pH=8.5). It was observed that the addition of the compound led to an increase in the electrical conductivity of the soil. The trace elements can be organized into two groups based on the results obtained in this study. One group contains the trace elements that were only present in the rows that were treated with the fertilizer. The other group of trace elements was mobilized throughout the whole plot.

**Conclusions** The application of sewage sludge on agricultural soils can be very useful as an organic amendment because it produces an increase in soil organic matter. However, sewage sludge must be applied with caution due to the changes in soil chemical properties (for example, pH and E.C.). The use of this type of waste for prolonged periods of time can cause problems of contamination in the soil.

**Keywords** Semiarid environment · Trace element · Vine · Waste

## 1 Introduction

The sludge generated in wastewater treatment plants can contain nutrients as it is organic in nature. One way to eliminate this waste is to use it as a fertilizer on agricultural soils. In fact, the use of sewage sludge in agriculture as an organic amendment is a practice that has increased in recent years. The use of fertilizer can alter the productivity of different crops and reduce the risk of nutrient leaching (Rigueiro-Rodríguez et al. 2012).

The sludge generated during the purification of wastewaters can contain compounds that are valuable to the soil (organic matter and phytonutrients, nitrogen, phosphorus or potassium (Bayo et al. 2010; Bonano et al. 2013)) and also compounds that are problematic in the environment (heavy metals and other contaminants, including cadmium, chromium, copper, mercury, nickel, lead, zinc and pathogens (Navarro

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2010)). These sludges are characterized by their high fluidity, and they have a heterogeneous composition, which varies depending on the origin of the wastewater and the environmental conditions (MMARM 2009). The removal of water, which represents 92–96 % of the total mass, from sludge leads to the concentration of other components (Navalon and Valor 2011). Prior to agricultural soil application, sludge may be stabilized and conditioned by treatment with reagents such as iron salts (ferric sulphate, ferric chloride), aluminium sulphate, sodium aluminates, lime and even polymers with active groups, which are used for exchange reactions (polyelectrolyte) (Elias 2009).

Numerous studies have been carried out on the use of sewage sludge as fertilizer. It is worth highlighting the review by Singh and Agrawal (2008) on the potential benefits and risks of land application. The review summarizes the main characteristics of sewage sludge, the effects of sewage sludge application on soil properties, the effects on growth, yield and heavy metal accumulation in several plants and the risks associated with sewage sludge amendment. Other studies provide an overview of the main methods for handling sludge and the technological options for their management (Fytily and Zabaniotou 2008; Wang et al. 2008; Smith 2009), with soil use recommended as the preferred management option when there is available land.

The agricultural use of sewage sludge is regulated and permitted in Spain by European bylaw Directive 86/278 (modified by Directive 219/2009). However, it was concluded in some studies that simple analysis of sewage sludge for heavy metal concentration in soils is not sufficient to correlate its risk, as the absorption of heavy metals varies with species and plant parts (Singh and Agrawal 2008).

In this context, the aim of the work described here was to consider whether changes in a vineyard soil after an extended period (over 8 years) of addition of sewage sludge as an organic amendment were reflected in the plant leaf composition, as suggested by several authors (Wild 1992; Higuera et al. 2012).

## 2 Materials and methods

The area under investigation was close to the municipality of Villarrubia de los Ojos (Ciudad Real). The proximity of this area to the ‘Tablas de Daimiel’ (an important Biosphere Reserve in Spain) (UNESCO 2014) means that the fundamental dynamics that govern these soils are related to the hydromorphism caused by the confluence of the rivers Guadiana and Cigüela. Furthermore, the soil rests on a limestone substrate (Jiménez Ballesta 2009). The area has an annual average temperature of 14.0 °C and an annual rainfall of 479.4 mm. In accordance with the Papadakis classification, the area has a ‘mild Mediterranean climate’ (MMA 2006).

The plot in which the experiment was carried out has an area of 3.5 ha, and it is dedicated to the cultivation of vines (variety Cencibel). Over the past 8 years, the plot had been fertilized with 25 t ha<sup>-1</sup> of a mixture of manure and sewage sludge (50 % of each component), and this treatment was applied at a depth of 30–40 cm. The fertilizer was added in alternate rows that coincided with the location of a drip irrigation system, with fertilizer and water always on the same side of the line.

The wastewater sludge was obtained from the sewage treatment station at Villarrubia de los Ojos, which produces a treated water volume of 6.489 m<sup>3</sup>/day. The characteristics of the sludge and the mixture applied as a fertilizer are shown in Table 1. It can be seen that the values comply with existing regulations in Spain for these compounds.

In this study, a total of six samples were taken from the surface horizon (Ap) of a plot where fertilizer (0.8 % moisture content) had been applied in alternate rows. Three samples were collected in rows where fertilizer had been applied and three samples in rows without fertilization. Another sample was taken from the surface horizon of a soil in an adjacent plot in which there had been no treatment (control sample).

The soil was described according to the criteria set out by the FAO (2006). Samples were taken to the laboratory where they were air-dried and sieved (<2 mm) prior to analysis. A sample of the sludge produced at the treatment plant in Villarrubia de los Ojos and a sample of the mixture used by the farmer, who was the owner of the plot, were also analysed.

**Table 1** Characteristics of the sludge produced by the Villarrubia WWTP, the mixture used in this study and legal limits according to Directive 86/278 (modified by Directive 219/2009)

Parameters	Sludge	Mixture	Legal limit
pH	6.3	6.6	Undefined legal limit
E.C.(dS/cm)	4.3	6.9	Undefined legal limit
CaCO <sub>3</sub> (%)	1.8	15.1	Undefined legal limit
O.M. (%)	34.0	31.5	Undefined legal limit
N (%)	4.1	0.8	Undefined legal limit
P (g/kg)	19.9	14.3	Undefined legal limit
K (g/kg)	4.3	11.7	Undefined legal limit
Ca (g/kg)	36.7	132.7	Undefined legal limit
Mg (g/kg)	8.2	10.6	Undefined legal limit
Na (g/kg)	1.36	2.8	Undefined legal limit
Fe (g/kg)	11.41	12.8	Undefined legal limit
Mn (mg/kg)	0.8	0.2	Undefined legal limit
Cu (mg/kg)	136.0	120.5	(1000–1750)
Ni (mg/kg)	42.2	13.7	(300–400)
Zn (mg/kg)	422.3	228.0	(2500–4000)
Pb (mg/kg)	26.3	19.9	(750–1200)
Cr (mg/kg)	48.2	22.1	(1000–1500)

E.C. electrical conductivity, O.M. organic matter

Leaf samples were also taken from the same areas from which the soil samples had been obtained. In each area, 20 leaves from the middle of the shoot were collected in September according to the methodology suggested by Ernst 1995. In the laboratory, the leaf samples were dried in an oven for 7 days at 36 °C.

The laboratory tests were conducted in accordance with the SCS-USDA (1972). In particular, the texture of the soil was determined using the densimeter method (Porta et al. 1986), and calcium carbonate was determined using a Bernard calcimeter (Porta et al. 1986). The organic matter was determined by potassium dichromate oxidation titration of remaining dichromate with ammonium ferrous(II) sulphate (Anne 1945).

The trace elements were studied by grinding samples in an agate mortar until the material had a diameter of less than 43 µm. The samples, both soil and leaves, were analysed by X-ray fluorescence on a Philips PW 2404 spectrophotometer with a maximum power of 4 kW (set of crystal analysers for LiF220, LiF200, Ge, PET and PX1, flow detector and twinkle detector). Analysis of the samples was carried out with pearls of lithium borate. Quality control was evaluated by duplicate analysis of certified reference samples (BCR 62, SMR 1573<sup>a</sup>, SMR 1515).

### 3 Results and discussion

The soil under investigation was classified as Haplic Arenosol (Calcaric, Novic) in accordance with the FAO-ISRIC-ISSS (2006) and as Typic Xeropsamment in accordance with Soil Taxonomy criteria (Soil Survey Staff 2006). The plot was dominated by a sandy class texture (88.3 %) (Table 2), and this implies good drainage, poor consistency and low fertility, as described previously for similar soils (Carlevaris et al. 1992). It was observed that the addition of the sludge did not affect the sand content in the soil.

The average results obtained for sampling points where treatment had been performed (CT) and the untreated sample points (ST) are given in Table 2; these data can also be compared with those obtained at the control plot (ZT). It can be seen that the treated area has a lower pH than the untreated

areas (ST), and this is due to the biodegradation of sewage sludge, which is rich in organic carbon (Singh and Agrawal 2010). Furthermore, it can be seen that the addition of the sludge led to an increase in the conductivity of the soil but did not cause salinization (Singh and Agrawal 2008; Roig et al. 2012); this increase in conductivity may be due to the use of salts to stabilize the sludge. In general, it can be stated that the application of this type of treatment modifies the chemical characteristics of the soil, as measured by the studied parameters, thus could increase the availability of heavy metals in the soil and their possible absorption into the plant as stated by Singh and Agrawal (2010).

The levels of major elements present in the samples are listed in Table 3. It can be seen that the average values for these elements in the treated soil ( $\bar{X}CT$ ) are higher than those found in the untreated soil ( $\bar{X}ST$ ), and in both of these cases, they are higher than those found in the control plot (ZT). Si, Al and Fe are in similar amount (they are the main constituents of mineral soils). From these results, it can be deduced that the application of this waste enriches the soil in major elements. The values obtained are lower than those published for calcareous soils of La Mancha (Amorós et al. 2012, 2013), which are also basic soils but are not as sandy. This finding is probably due to the fact that sandy soils do not retain elements efficiently (Wild 1992). The treated soils have lower contents of Na, Mg, Al, K, Mn and Fe and higher values of Si, P, S and Ca than world soil references (Kabata-Pendias 2001; Sparks 2003). High content of Si is due to the class texture (sandy).

On studying the trace elements (Table 4), a similar trend was observed to that found for the major elements. The total concentration of heavy metals in soils depends directly on the soil type and indirectly on the pH (Singh and Agrawal 2008; Smith 2009). Due to the pH and the sandy texture of the studied soils, the levels of elements retained are very low, and all values are lower than those found for the limestone soils of Castilla-La Mancha (Amorós et al. 2012) and global values (Kabata-Pendias 2001; Sparks 2003).

The results indicate that, although the fertilizer was applied in a localized area in the plot, the components can flow across the land both in terms of depth and laterally. This mobility is evidenced by the fact that the values obtained

**Table 2** Chemical characteristics (mean±standard deviation)

	pH	E.C. (dS/m)	CaCO <sub>3</sub> (%)	Sand	O.M. (%)	C/N	P <sub>2</sub> O <sub>5</sub> (mg/kg)	C.E.C (cmol <sup>+</sup> )	BS(%)
$\bar{X}CT$	8.0±0.3	0.7±0.5	4.9±0.7	89.0±0.4	2.2±0.2	12.1±0.4	22.1±6.1	10.3±0.6	100±0.0
$\bar{X}ST$	8.5±0.1	0.1±0.1	4.2±0.3	87.5±0.0	1.2±0.1	11.7±0.5	11.3±4.8	15.3±1.2	100±0.0
ZT	8.1	0.5	13.7	84.8	0.5	10.9	3.5	16.3	100

E.C. electrical conductivity, O.M. organic matter, C.E.C. cation exchange capacity, BS(%) base saturation,  $\bar{X}CT$  average for treated areas,  $\bar{X}ST$  average for untreated areas, ZT control plot

**Table 3** Values for major elements in g/kg (mean±standard deviation)

	$\bar{X}CT$	$\bar{X}ST$	ZT	CLM <sup>a</sup>	World <sup>b</sup>
Na	0.68±0.05	0.63±0.10	0.58	1.3	5.0
Mg	2.38±0.22	1.80±0.21	1.71	17.7	5.0
Al	17.6±1.65	17.44±1.61	15.73	81.1	71.0
Si	403.98±8.08	413.00±6.90	402.68	292.0	330.0
P	1.00±0.43	0.52±0.07	0.45	1.9	0.80
S	1.57±1.33	0.30±0.07	0.33	2.4	0.70
K	10.06±1.17	9.93±1.17	8.58	15.4	15.0
Ca	21.18±4.42	19.10±4.50	29.00	301.0	15.0
Mn	0.44±0.50	0.12±0.02	0.15	0.4	1.0
Fe	5.57±0.91	5.71±0.58	5.35	25.6	40.0

$\bar{X}CT$  average for treated areas,  $\bar{X}ST$  average for untreated areas, ZT control plot

<sup>a</sup> CLM (Castilla-La Mancha) levels of calcareous soil (Amorós et al. 2013)

<sup>b</sup> Levels for world soils calculated as the average from references (Kabata-Pendias 2001; Sparks 2003)

in the treated areas are higher than those obtained in untreated areas of the plot, which in turn are higher than those for the control plot.

It can be seen that the trace elements show different behaviour in the soil, and these can be placed into two groups. The first group of elements consists of those that are more concentrated in the treated area (Ni, Zn, Sr, Nb, Pb and Ba). The

**Table 4** Values of trace elements in mg kg<sup>-1</sup> (mean±standard deviation)

	L.D. <sup>a</sup>	$\bar{X}CT$	$\bar{X}ST$	ZT	CLM <sup>b</sup>	World <sup>c</sup>
V	1.61	15.4±1.03	14.8±2.04	13.9	32.9	95.0
Cr	0.90	27.5±0.40	34.2±6.66	28.8	32.6	68.5
Co	1.91	2.6±0.25	2.3±0.46	3.0	6.8	9.0
Ni	0.81	28.3±22.06	14.9±2.95	14.6	15.8	37.0
Zn	0.56	28.3±11.69	17.7±2.21	15.5	32.3	78.5
Sr	0.20	79.0±15.02	46.6±10.38	48.0	299.0	220.0
Nb	0.28	5.9±0.64	5.7±0.61	5.6	9.4	13.0
Cs	0.11	1.1±1.62	1.00±1.62	2.5	2.5	3.5
Ba	3.86	104.6±27.05	94.8±15.06	76.2	175.0	513.0
Ce	1.33	18.4±2.29	18.7±4.60	15.2	36.6	55.0
Pb	0.06	11.3±1.17	9.9±1.37	9.3	17.6	32.0
Nd	3.59	11.4±1.35	9.7±1.60	10.1	15.9	30.5

$\bar{X}CT$  average for treated areas,  $\bar{X}ST$  average for untreated areas, ZT control plot

<sup>a</sup> Detection limit of the equipment used in the determination of trace elements

<sup>b</sup> CLM (Castilla-La Mancha) levels for calcareous soil (Amorós et al. 2013)

<sup>c</sup> Levels for world soils calculated as the average from references (Kabata-Pendias 2001; Sparks 2003)

second group of elements remained relatively constant over the whole area (V, Co, Cs and Ce). In the treated rows, Ni, Zn and Pb have higher values than in the untreated rows and the control plot (albeit never above legal limits). The case of chromium warrants special attention as this is an element that is widely used in the stabilization of the sludge but is found in larger amounts in the untreated rows (34.2 mg/kg) than in the treated rows (27.5 mg/kg).

The bioaccumulation of elements in the leaves of the vine was evaluated by calculating the Biological Absorption Coefficient (BAC). The BAC is the ratio between leaf concentration and soil concentration (Kabata-Pendias 2001). The total soil value was calculated as the average value for the six soil samples (Table 5). Although metals are retained in the soil, their uptake by plants depends on the soil type (Soriano-Disla et al. 2014). Very few world references were found regarding the concentration of trace elements in vine leaves (Amorós et al. 2013). The highest bioaccumulation values were obtained for caesium and strontium, which are the two elements used in flocculation and sludge stabilization. These values are below the literature toxicity limit for human health (Higuera et al. 2012).

Comparison of the values obtained with those reported globally for different plants (Kabata-Pendias 2001) allows the following conclusions to be drawn:

**Table 5** Biological Absorption Coefficient (BAC)

	$\bar{X}TS$	$\bar{X}H$	BAC
Na	0.65±0.04	0.04±0.05	0.08
Mg	2.09±0.42	3.96±0.78	1.90
Al	17.52±0.11	0.87±0.07	0.05
Si	408.5±6.37	14.89±2.35	0.04
P	0.76±0.33	1.03±0.16	1.35
V	15.1±0.4	5.4±1.8	0.4
Cr	30.9±4.7	6.1±0.2	0.2
Co	2.4±0.2	1.8±0.3	0.8
Ni	21.6±9.5	1.0±1.8	0.02
Zn	21.1±4.8	14.3±1.0	0.7
Sr	62.7±23.0	192.8±33.5	3.1
S	0.93±0.90	1.95±0.22	2.09
K	10.0±0.09	8.31±1.80	0.83
Ca	20.14±1.47	22.56±3.62	1.12
Mn	0.28±0.22	0.09±0.03	0.32
Fe	5.64±0.09	0.23±0.03	0.04
Nb	5.8±0.1	4.1±0.1	0.7
Cs	1.1±0.8	4.9±2.1	4.5
Ba	99.7±7.0	36.3±1.9	0.4
Ce	18.2±0.2	7.8±3.8	0.4
Pb	10.6±1.0	3.2±0.1	0.3
Nd	10.6±1.36	1.8±0.5	0.5

$\bar{X}TS$  average for six soils,  $\bar{X}H$  average for leaves

- Values greater than or close to 1: The values obtained were S (2.09, often used in pesticides), Mg (1.90), P (1.35) and Ca (1.12) for the major elements and Cs (4.5) and Sr (3.1) for the trace elements.
- Values between 0.1 and 1 were obtained for K (0.83) of the major elements and Nb (0.7), Zn (0.7), Nd (0.5), V (0.4), Ba (0.4), Ce (0.4), Cr (0.4) and Pb (0.3) of the trace elements.
- Values below 0.1 were obtained for Na (0.08) and Si (0.04) of the major elements and Ni (0.02) of the trace elements

It can be stated that certain elements (such as Al, Si, K, S and Cr) accumulate in the plant at similar levels when compared with other species (Kabata-Pendias 2001) and other vines (Amorós et al. 2013). On the other hand, it must be highlighted that the majority of the studied elements (Mg, K, Ca, V, Co, Sr, Nb, Cs, Ba, Ce and Nd) are accumulated in the vines in this work at higher levels than in other vineyards (Amorós et al. 2013) and other species (Kabata-Pendias 2001). Although the levels of elements retained in the soil are very low due to its sandy texture, the BAC indicates that the uptake of the elements by the plant is higher than in vineyard soils amended with other wastes (Pérez-de-los-Reyes et al. 2013).

#### 4 Conclusions

The use of sewage sludge led to the modification of certain chemical soil properties. These modifications included a decrease in pH (from 8.5 to 8.0), an increase in electrical conductivity (0.1 to 0.7 dS/m) and an increase in the amount of organic matter (1.2 to 2.2 %).

With regard to major elements, it can be stated that the application of this waste enriches the soil in these elements, although the sandy soils do not retain them efficiently. On studying the trace elements, we observed a similar trend to that found for the major elements. There are two groups of trace elements with respect to their mobility in the soil. Chromium is a special case because it is found in large amounts in untreated rows (34.2 mg/kg).

Comparison of the BAC values for different types of vineyards and soils showed that the majority of the studied elements in our work are accumulated at higher levels than in other calcareous vineyard soils. The BAC indicates that the uptake of the elements by the plant is higher in sandy vineyard soils than in those amended with other wastes.

The long-term application of sludge can lead to the contamination of soil. In the case reported here, the low retention capacity of sand for pollutants means that contaminants could reach the groundwater in sensitive areas such as ‘Tablas de Daimiel’.

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