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# Chapter

# Sewage Sludge to Fertilise Durum Wheat: Effects on Crop and Soil

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# **Abstract**

The vast quantities of degradable waste generated in urban areas may negatively influence the environment if improperly managed. This study examines effects on soil properties, yield and morphological performance of winter wheat (Triticum turgidum L. cv. Vitron) after applying composted and air-dried sewage sludge. The experiment was conducted on the field scale in two different farm soil plots Toledo, (central Spain) with different characteristics, especially salinity, concentration of chlorides, sulphates and pH. Three fertiliser treatments were considered: commercial fertiliser; air-dried sewage sludge and composted sewage sludge. Sewage sludge promoted better yields than the commercial fertiliser, and preserved soil physicochemical characteristics. The sewage sludge application (air-dried and composted) to soil improved the results of the morphological characteristics of the studied wheat in relation to the commercial fertiliser. In the saline soil plot, air-dried sewage sludge improved the morphological characteristics of spikes (length, weight, number of grains per spike), but not final grain weight and, consequently, yield. These were upgraded with the composted sewage sludge. Use of sewage sludge for winter wheat production was the better studied option and proved a sustainable approach to recycle such waste on land.

Keywords: sewage sludge, compost, wheat, spike, soil, reuse, sustainability

#### 1. Introduction

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The rising prices of mineral fertilisers have led more farmers to consider organic matter to cover crop nutrient requirements and to maintain soil fertility [1]. It is possible to adequately process organic urban waste and obtain organic fertilisers following aerobic/anaerobic treatments [2]. Aerobic organic waste transformation leads to a well-humified stabilised material (compost) that can be used as fertiliser because it comprises many essential plant nutrients: P, N, Zn, K, Mn, Cu, Fe, etc. Organic matter from biosolids is an important source of essential nutrients for agricultural crop growing [3]. It enhances soil physico-chemical properties, encourages soil activity and microbial growth, and promotes good soil structure which, in turn, improves water holding capacity and aeration. However, this practice poses potential risks related to the accumulation of contaminants in surface soil. Nevertheless, benefits from applying biosolids to farmland have been well documented [4–6], and are fostered by European legislation currently in force; e.g. Council Directive 86/278/EEC on Sewage Sludge [7], which is believed to be the best environmental practice option for most circumstances [8].

Sewage sludge is a by-product from wastewater treatment processes. Given the increases in wastewater treatment plant activity and quantity of produced waste materials, waste management has become a real environmental problem [9]. Fortunately, orientating this waste to agricultural recovery is a management strategy that forms part of sustainable development [10–12]. Its land application is a relevant incentive as regards nutrient recycling, reuse and soil amendment [13, 14], and the fertilising effect has been observed more for well-drained soil [15]. Sewage sludge nutrient content sustains soil fertility, while soil properties are improved by organic constituents. Using sludge in agriculture is one of the solutions that the European Union contemplates for this waste and is considering circular economy practice in which waste becomes a resource.

However, sewage sludge, like other biosolids, can contain large quantities of toxic heavy metals (e.g., Cr, Pb, Ni, Hg and Cd) because industrial wastewater is mixed with sewage. The disagreement about the agricultural application of sewage sludge is related to the quality and safeness of food products [16]. Being sustainable does not mean that it does not incur risks associated with the characteristics of the material itself or with its handling. Those risks must be controlled so they do not have any significant negative effects. The way to do this is to study each case individually because no standard sewage sludge composition exists.

Applying metal-polluted sewage sludge can mean crop damage, soil/water pollution and heavy metal accumulation in the food chain. The magnitude of this problem lies in the composition of sludge and its application rate, crop species and management practices, as in soil properties [14].

Most potential toxicity problems lie in biological toxicity tests, which allow us to observe how a certain toxic or contaminated agent impacts the viability of a living being. Ecotoxicity is calculated by measuring  $EC_{50}$ , which is the dose of the compound required for half a population (bacterial in this case) to die. To this end, a leachate is obtained that simulates the transfer of contaminants, which takes place in the medium when it encounters water [17].

Using compost from sewage sludge and organic municipal solid waste in agriculture offers the potential for recycling plant nutrients and to, thus, reduce the employment of mineral fertilisers. Previous studies have confirmed that compost fertilisers enhance microbiological soil physico-chemical properties and enlarge the pool of nutrients, such as P [18] and organic C [14, 19] in soil. Moreover, when composting sewage sludge, health hazards from pathogens (bacteria, protozoa, parasitic helminths, etc.) are reduced [12].

It has been proved than sewage sludge-based fertilisation might be the most effective way to mitigate the negative effects of water stress on wheat yield in arid and semiarid regions [11], and also a good form of substitution towards environmentally friendly agriculture.

Using sewage sludge as a fertiliser has led to promising results in agriculture compared to chemical fertilisers [11, 12, 20].

Moreover, wheat is one of the world's most widely grown crops. In 2019/2020, the European Union produced a wheat volume that came to 153.5 million metric tons [21]. Durum wheat possesses excellent food qualities, namely minerals, gluten and high-fibre content. In Spain, an average of 6 million hectares of cereals is cultivated. It is the sector with the largest territorial base and with nation-wide distribution. The 2018 national cereal harvest (marketing year 2018/19) is estimated at 23.26 million tons, 44.45% more than the previous year, and was characterised by a bad season due to drought. In autumn-winter cereals, 28.9% correspond to soft wheat with 6.7 million tons, and 5.7% to durum wheat with 1.32 million tons [22].

Spain could become the 5th community producer with 23.1 Mt. According to the Junta de Comunidades de Castilla-La Mancha (JCCM) [23], wheat production in Castilla-La Mancha (Spain) was 542,000 tons in 2017.

Wheat (*Triticum aestivum* L.) has a prominent place among cereals for its high nutritional value. Its grain contains starch (60–68%), protein (6–21%), fat (1.5–2.0%), cellulose (2.0–2.5%), minerals (1.8%) and vitamins [24]. It is one of the most important grain crops grown all over the world and plays a major role in the economic activity of most countries [25]. Durum Wheat (*Triticum durum*) cultivation is very ancient in semiarid Mediterranean areas [11]. With winter wheat, obtaining high grain yield depends on sufficient numbers of fertile spikes and enough shoot biomass developing [26]. To achieve these objectives, fertiliser composition must be considered and, thus, fertilisers produced from sewage sludge can be a suitable solution.

When we talk about sewage sludge, we usually generalise something that is extremely variable. Sewage sludge is not all the same in composition and danger terms as some hardly contain pollutants and can even be used directly. Others, however, possess many dangerous and polluting components. Our study focused on that produced in a specific place.

The main aim of this field trial was to assess the response of crop and soil that underwent three different fertiliser treatments based on a one-time waste input from a wastewater treatment plant (WWTP). We hypothesised that WWTP waste (composted or air-dried sewage sludge) as the initial fertility driver of soil amendments would enhance soil physico-chemical properties and final durum wheat yields. The study objectives were to investigate the effect of applying: (i) composted sewage sludge, air-dried sewage sludge and commercial fertiliser on the morphological characters of wheat crop (spike and grain) and yield; (ii) the above fertiliser treatments on soil characteristics in relation to the original soil.

#### 2. Materials and methods

#### 2.1 Study area and experimental design

Field trials were run in two dry-land agricultural plots (15 ha each) located in the province of Toledo (central Spain): Villacañas (39°35′17" N; 3°27′45"W) and Quero (39° 33′ 7.16" N; 3° 15′ 37.17" W). These plots have been historically cultivated for winter cereals (oat, barley, rye, or wheat) and sporadically intercalated with fallow. A local weather station (YUTM 4340164, XUTM: 482750, altitude 658 m) reported the mean minimum, mean maximum and mean average temperatures, which were respectively  $-0.46^{\circ}$ C, 25.56°C and 10.93°C. Total precipitation was 275.5 mm.

Three fertiliser treatments were considered: commercial fertiliser (CF); airdried sewage sludge (SS); composted sewage sludge (CS). We opted to use 15 t ha<sup>-1</sup> in all three cases because it is the usually applied dose in the local area for durum wheat when using this CF (8–16-8). Considering the composition of SS and CS (**Table 1**), the equivalence in terms N-P-K were SS (6–5-0.3) and CS (4.3–5.3-0.5).

Replication is the most important aspect of an experimental design [27, 28]. Hence each test plot (Villacañas and Quero) was divided into 27 subplots (9 replicates per fertiliser treatment: CF, SS and CS) that were randomly distributed.

Plots were prepared using farm machinery early in December and treatments (CF, SS and SC) were applied to soil. Next 180 kg ha<sup>-1</sup> of *Triticum turgidum* L. cv. Vitron seeds were sown 2 weeks later. This is a high-yielding durum wheat variety characterised by medium earliness in spike emergence, a light to dark brown speak colour, medium plant height and being well-adapted to all growing regions.

# 2.2 Characteristics of experimental materials

Before conducting the experiment, the physico-chemical parameters of both air-dried and composted sewage sludge were determined (**Table 1**).

# 2.2.1 Air-dried sewage sludge

Sewage sludge was obtained from the Alcázar de San Juan WWTP (29,000 inhabitants, central Spain: 391 240 N; 31 120 W. Altitude, 644 m). This WWTP purifies wastewater by a biological activated sludge system. This wastewater is a mixed kind (70% domestic, 30% industrial) and comes from two industrial areas [29] related to winemaking and cheese-making. **Tables 1** and **2** show its chemical characterisation. Organic compounds (PAHs and PCBs), *Salmonella* sp., *E. coli* and ecotoxicity were also determined.

Agronomic Parameters	Air-dried sewage sludge	Composted sewage sludge
рН	7.01	5.830
Moisture (%)	86.46	9.28
Electrical conductivity (μS cm <sup>-1)</sup>	3087	5740
Total organic matter (%)	65.25	52.08
Total N (%)	5.95	4.30
P <sub>2</sub> O <sub>5</sub> (%)	6.10	5.47
K <sub>2</sub> O (%)	0.31	0.48
CaO (%)	6.78	12.53
MgO (%)	0.87	1.04
Fe (mg kg <sup>-1</sup> d.m.)	50688	10301
C:N ratio	5.95	6.68

**Table 1.**Characteristics of the air-dried and composted sewage sludge. d.m: dry matter.

Parameter (mg kg <sup>-1</sup> )	Soil Villacañas	Soil Quero	R.D. 1310/90 value limit for soil		Air-dried sewage sludge	Composted sewage sludge	R.D. 1310/ limit for slu	sewage
			Soil with pH < 7	Soil with pH > 7			Soil with pH < 7	Soil with pH > 7
Total Cd	0.1	<0.10	1	3.0	0.33	2.38	20	40
Total Cu	<1.00	<1.00	50	210.0	189	116	1	1,75
Total Cr	15.51	2.21	100	150.0	24.6	26.3	300	400
Total Hg	<0.10	<0.10	1	1.5	30.1	29.9	750	1,2
Total Ni	11.37	1.5	30	112.0	512	431	2,5	4
Total Pb	11.30	11.30	50	300.0	0.45	<0.2	16	25
Total Zn	<20.00	<20	150	450.0	30.9	35.1	1	1,5

**Table 2.**Metal content in original soils, air-dried sewage sludge and composted sewage sludge.

In view of the analytical results (**Tables 1** and **2**), waste had pH 7.01, a high moisture content (over 80%), showed high electrical conductivity (3,087  $\mu$ S cm<sup>-1</sup>) and was rich in organic matter (nearly 70%). The total N (5.95%), P (6.10%) and K (0.31%) contents had an interesting agronomic value because of their fertilising potential. The N content and C:N ratio (5.95) indicated that this waste could be used for composting as a nitrogen source.

No performed analysis detected *Salmonella* sp., although *E. coli* (1.4 10<sup>5</sup> colonyforming units, cfu) was found. According to Spanish Royal Decree 1310/90 [30], metals did not exceed the legal limits (**Table 2**). Otherwise organic compounds are limited by a European Union Directive. According to this legislation, the sum of 16 PAHs (Naphtalene, Acenaphthylene, Acenaphthene, Fluorene, Phenanthrene, Anthracene, Fluorabthene, Pyrene, Benz (a) anthracene, Chrysene, Benzo (b) fluoranthene, Benzo (k) fluoranthene, Benzo (a) pyrene, Indeno, Benzo (g.h.i.) perylene and Dibenz (a.h) anthracene) must to be below 6 mg kg<sup>-1</sup> dry matter and the sum of the seven PCBs (PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153, PCB 180) cannot exceed 0.8 mg kg<sup>-1</sup> dry matter. The content of PAHs and PCBs in the studied air-dried sewage sludge was less than 10 μg kg<sup>-1</sup> dry matter and were, therefore, lower than the EU Directive limits.

As the purposes of using sewage sludge was an agriculture application, an ecotoxicity test was carried out to estimate its potential post-application risk [31]. According to Spanish law [32], waste is characterised as ecotoxic and is, therefore, dangerous when the Effective Concentration 50 (EC<sub>50</sub>) value is 3,000 mg L<sup>-1</sup>. In view of the analysis results (502,700 mg L<sup>-1</sup>), it can be stated that the Alcázar de San Juan WWTP sludge is not considered ecotoxic waste.

# 2.2.2 Composted sewage sludge

The sewage sludge from the Alcázar de San Juan WWTP was composted by a wind-row composting system. Cereal straw (80% dry matter: 4.2% crude protein, 73% fibre (cellulose, hemicellulose and lignin), 0.35% Ca, 0.15% and P, 2.36% K) was used as a carbon source and also for helping to improve the compost structure thanks to its fibrous texture. As the sewage sludge required being dried beforehand, it was spread over an area of the composting plant before being incorporated. The downwards to upwards order of the materials for pile formation was straw plus sludge plus straw. The composting ratio was two parts straw to one part sludge (v/v). The straw and sludge mixture were periodically flipped to homogenise it and to improve the ventilation of piles. Straw was applied without grinding, which not only provided the mixture with C, but also helped to improve the compost structure. The composting process was completed 4 months after starting to dry sewage sludge. **Tables 1** and **2** provide the final composition details.

High values of electrical conductivity (5,740  $\mu$ S cm<sup>-1</sup>) and Fe (10,301 mg per kg of dry matter) were detected. CaO (12.53%) and MgO (1.04%) concentrations were higher than that of the air-dried sewage sludge (6.78% and 0.87%, respectively).

Moisture was 9.28%. The C:N ratio after composting was 6.68 (**Table 1**), so it was a fairly mineralised compost. It had high organic matter (52.08%) and total N (4.30%) contents. The concentration of metals (**Table 2**) was lower than that allowed by law. It was not ecotoxic (<2 toxicity units) and was a microbiologically stable compound. Both *Salmonella* sp. and *Escherichia coli* < 1,000 c.f.u g<sup>-1</sup> were absent.

#### 2.2.3 Commercial fertiliser

NPK 8–16-8 + organic matter (22%) was the commercial fertiliser employed in the control subplots. This fertiliser is frequently used by farmers in the area. Further

details from the label are:  $P_2O_5$  (soluble in neutral ammonium citrate and water), 14.50%;  $P_2O_5$  (soluble in water), 11.50%;  $K_2O$  (soluble in water), 8.00%; Organic C, 12.50%; Humic acids, 1.00%; Cu, 208 mg kg<sup>-1</sup> dry matter; Zn, 559 mg kg<sup>-1</sup> dry matter.

#### 2.3 Sampling and measurement

#### 2.3.1 Soil

Initially a soil sample from each study plot (Villacañas and Quero) was collected at 10 different random points in an "S" pattern at a depth of 0–35 cm with a hand auger. The analysed parameters were EC, Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, organic matter, N, C:N ratio, P, CO<sub>3</sub><sup>2-</sup>, K:Mg ratio, Ca:Mg ratio, Fe, Zn, Cu, Mn, B, cation exchange capacity (CEC), K, Na and Ca (**Table 3**). One month after harvesting, a 0–35 cm-deep soil sample was collected from 10 random points of each replicate. Therefore, nine soil samples per treatment (CF, SS and CS) from each test plot (Villacañas and Quero) were collected and analysed. The assessed parameters are presented in **Table 3**.

Parameter	Units	Villacañas	Quero
рН		8.60	9.15
EC	mmhos cm <sup>-1</sup>	0.52	13.65
Cl <sup>-</sup>	mg kg <sup>-1</sup>	42	153
SO <sub>4</sub> =	mg 100 <sup>-1</sup> g	75	2102
Total CO <sub>3</sub> <sup>=</sup>	%	30,5	15.60
Org. matter	(%)	1.69	2.22
Total -N	%	0.09	0.10
Nitric-N	${\rm mgkg^{-1}}$	5.0	19
Soil-N	kg ha <sup>-1**</sup>	19.23	73
Ass. P	mg kg <sup>-1</sup>	19	9
Ass. Fe	mg kg <sup>-1</sup>	2.72	0.35
Ass. Zn	mg kg <sup>-1</sup>	0.27	0.38
Ass. Cu	mg kg <sup>-1</sup>	0.78	0.35
Ass. Mn	mg kg <sup>-1</sup>	8.70	2.15
Ass. B	mg kg <sup>-1</sup>	0.61	2.45
Ass. K	meq 100 <sup>-1</sup> g	0.66	0.77
Ass. Na	meq 100 <sup>-1</sup> g	1.70	3.80
Ass. Ca	meq 100 <sup>-1</sup> g	20,31	87.14
CEC	meq 100 <sup>-1</sup> g	12.3	10
C:N ratio		11	13
K:Mg ratio		0.60	0.04
Ca:Mg ratio		18.5	4.9

<sup>\*\*</sup>N-soil was calculated according to the equation N-soil =  $(N-nitric\ x\ 50)/(10\ x\ density)$ , where N-nitric is expressed as mg kg<sup>-1</sup> and density is 1.

**Table 3.**Characterization of Villacañas and Quero initial soil plots. Ass: assimilable. CEC: Cationic Exchange Capacity.

Each soil sample was air-dried, sieved to <2 mm and analysed by the following techniques: texture by the Bouyoucos hydrometer method; pH by the potentiometric method (saturated soil paste 1:2,5); electrical conductivity; N by the Kjeldahl procedure; extractable P. Soil samples were prepared for the analysis with acid digestion to determine Ca, Mg, Na and K (atomic emission); Fe, Zn, Cu, and Mn by atomic absorption spectroscopy.

# 2.3.2 Crop

The preharvested random (215 post-sowing days) bunches of plants were cut far away from the bottom, close to soil, at the center of the subplot (replicate) to prevent board effects. Compared to soil sampling, nine sets of collected plants were used (1 sample x 3 treatments x 3 replicates) per treatment. Yield information (kg ha<sup>-1</sup>) was recorded. Finally, in the laboratory, 25 plants from each set sample were chosen to estimate spike weight, and length, number, and total weight of grains per spike.

# 2.4 Statistical procedure

The experimental design involved three treatments (CF, SS and CS) and nine replicates per plot. The data about spike weight (g), spike length (cm), number of grains per spike and total weight of the set of grains of that spike (g) were subjected to statistical processing by an analysis of variance. This was done to determine the factor or factors, and the possible interactions between them, which could affect any differences observed among the treatments.

All the statistical calculations were performed with Statgraphic Centurion XV. The ANOVA table decomposes the variability of the studied parameters (spike length, spike weight, number of grains per spike, total weight of grains per spike and yield) in contributions due to several factors. In the analysis, the sum of the type III squares was chosen. Thus, the contribution of each factor was measured by eliminating the effects of the other factors. P-values proved the statistical significance of all the factors, and those less than 0.05 indicated that these factors had a statistically significant effect on each parameter at the 95% confidence level.

#### 3. Results

#### 3.1 Crop

**Table 4** displays the effect of the factors plot (Villacañas or Quero), treatment (SS, CS and CF) and plot-treatment interaction on the studied parameters. Spike length, spike weight, and number of grains per spike were significantly influenced by the plot where the crop was grown (Villacañas or Quero), and by treatment (CF, SS or CS). This did not occur with weight of grains per spike and, consequently, with yield. The plot-treatment interaction was not significant in any case.

A simple factorial ANOVA based on the treatment received (**Table 5**) showed that the type of treatment in Villacañas affected all the studied parameters, except for spike weight.

In Quero, only spike length and spike weight were influenced by treatment. No significant differences were observed in either number of grains per spike or weight of grains or yield.

FACTOR	Spike length	Spike weight	Number of grains per spike	Weight of grains per spike	Yield
PLOT (A)	0.0000*	0.0000*	0.0000*	0.0972	0.0972
TREATMENT (B)	0.0000*	0.0131*	0.0329*	0.0594	0.0594
A-B	0.2607	0.1613	0.1568	0.0672	0.0672
Significant differences					

Table 4

P-values for plot (A): Villacañas, Quero; treatment (B): CF, CS, SS; interaction (A)-(B) for average spike length, average spike weight, average number of grains per spike, average total weight of grains per spike and average yield.

# 3.1.1 Average spike length

In Villacañas (**Table 5**), no significant differences appeared between SS and CS, and these treatments led to a somewhat longer length (5.47 cm and 5.51 cm, respectively) than with CF (5.09 cm).

In Quero (**Table 5**), spike length was longer than that obtained in Villacañas for all the treatments, and was significantly longer than that achieved by applying SS and CS (6.22 cm and 6.12 cm, respectively) in relation to that achieved when applying CF (5.95 cm).

# 3.1.2 Average spike weight

In Villacañas (**Table 5**), a statistically significant increase in the average spike weight was observed in the plants grown with SS (1.41 g) compared to those grown with CS (1.25 g) or CF (1.28 g).

In Quero (**Table 5**), like spike length, spike weight was also heaiver than in Villacañas. Weight improved with SS (1.68 g) and with CS (1.66 g) compared to the plants grown with CF (1.49 g).

#### 3.1.3 Average number of grains per spike

In Villacañas (**Table 5**), similarly to average spike weight, the average number of grains per spike was SS (20 grains) than in those plots where CF or CS was applied (18 and 17 grains, respectively).

In Quero (**Table 5**), the same occurred for this parameter as in Villacañas. It was in the subplots with SS and CS where the number of grains per spike was significantly bigger (24) than for the other treatment CF (22). Likewise, it was noteworthy that the number of grains per spike in the Quero plot was always bigger than that obtained in the Villacañas plot, where 20 grains per spike were never exceeded.

# 3.1.4 Average total weight of grains per spike

In Villacañas (**Table 5**), and following the same line as the previously mentioned parameters, the average total weight of grains per spike was significantly heavier (0.84 g) in the subplots with SS than in those where CS (0.72 g) or CF (0.75 g) was applied.

In Quero (**Table 5**), barely any differences were observed with the Villacañas plot, a trend observed of a slightly increased total weight of grains per spike. In this plot with the compost application, a statistically significant heavier average total weight of grains per spike was recorded (0.86 g).

	Treatment	Average spike length (cm)	Average spike weight (g)	Average number of grains per spike	Average total weight of grains per spike (g)	Average yield (kg ha <sup>-1</sup> )
Villacañas	P-value	0.0002*	0.0099	0.0376*	0.0336*	0.0336*
	CF	5.09 ± 0.75a	1.28 ± 0.56a	18 ± 7a	0.75 ± 0.33a	2994 ± 1337ab
	SS	5.47 ± 0.88b	1.41 ± 0.60b	20 ± 7b	0.84 ± 0.37b	3371 ± 1481b
	CS	5.51 ± 0.85b	1.25 ± 0.60a	17 ± 7a	0.72 ± 0.37a	2884 ± 1476 a
Quero	P-value	0.0269*	0.0166*	0.1497	0.1297	0.1297
	CF	5.95 ± 0.70a	1.49 ± 0.50a	22 ± 7a	0.76 ± 0.36a	3056 ± 1437a
_	SS	6.22 ± 0.76b	1.68 ± 0.47b	24 ± 7b	0.83 ± 0.33ab	33171 ± 1311ab
_	CS	6.12 ± 0.78ab	1.66 ± 0.53b	24 ± 7a	0.86 ± 0.34b	3423 ± 1357b

<sup>\*</sup>Significant differences.

#### Table 5.

P-values and average  $\pm$  standard deviations of average spike length (cm), average spike weight (g), average number of grains per spike, average total weight of grains per spike (g) and average yield (kg ha<sup>-1</sup>). Distinct letters mean groups with significant differences at P < 0.05 among treatments for the same study plot according to the LSD test.

#### 3.1.5 Yield

The maximum yield was obtained in the Quero plot (3,423 kg ha<sup>-1</sup>) with the minimum one in Villacañas (2,884 kg ha<sup>-1</sup>), both cases with CS (**Table 5**). It was, therefore, clear that soil type strongly influenced crop response to the application of the same product. In Villacañas, the highest yield was obtained with SS (3,371 kg ha<sup>-1</sup>) and the lowest with compost (2,884 kg ha<sup>-1</sup>). On the contrary, CS treatment gave the highest yield in the Quero plot (3,423 kg ha<sup>-1</sup>), where the lowest yield was accomplished with CF treatment (3,056 kg ha<sup>-1</sup>).

#### 3.2 Soil

The initial Villacañas test soil plot (**Table 3**) had a sandy loam texture [33], basic pH (8.6) and was somewhat saline. The contents of Cl<sup>-</sup> (42 mg kg<sup>-1</sup>), CEC (12.3 meq  $100^{-1}$  g), Fe (2.72 mg kg<sup>-1</sup>), Zn (0.27 mg kg<sup>-1</sup>) and Cu (0.78 mg kg<sup>-1</sup>) were medium, while the levels of organic matter (1.69%) and total N (0.09%) were low [34]. Normal N release was indicated by the C:N ratio (11) [35]. Finally, the amounts of P (19 mg kg<sup>-1</sup>), total CO<sub>3</sub><sup>2-</sup> (30.5%), K (0.66 meq  $100^{-1}$  g), Na (1.70 meq  $100^{-1}$  g) and Mn (0.70 mg kg<sup>-1</sup>) were high [34].

Soil from the Quero test plot (**Table 3**) had higher pH (9.15) than the Villacañas one, but the difference lay in a very saline soil. The contents of Cl<sup>-</sup> (153 mg kg<sup>-1</sup>) and K (0.77 meq  $100^{-1}$  g) were high. Na (3.80 meq  $100^{-1}$  g) and Ca (87.14 meq  $100^{-1}$  g) came in large amounts. Organic matter content (2.22%), total  $CO_3^{2-}$  (15.63%), CEC (10 meq  $100^{-1}$  g), Zn (0.38 mg kg<sup>-1</sup>), Cu (0.35 mg kg<sup>-1</sup>) and Mn (2.15 mg kg<sup>-1</sup>) were medium [34]. The C:N ratio indicated little N release [35].

The main difference between the original soils of both plots were salinity and  $SO_4^{2-}$  content as the Quero plot soil was very saline (EC: 13.65 mmhos cm<sup>-1</sup>) and rich in gypsum (2,102 mg  $100^{-1}$  g), unlike the Villacañas soil (EC: 0.52 mmhos cm<sup>-1</sup> and 75 mg  $100^{-1}$  g, respectively).

In the soils to which CF was applied significant differences were observed between Villacañas and Quero for all the analysed parameters (**Table 6**), except for Cu, Mn and CEC.

In the Quero plot soil (**Table 4**), the values of pH (9.14), EC (19.86 mmhos cm<sup>-1</sup>) and the C:N ratio (6.37), and the contents of Cl<sup>-</sup> (286 mg kg<sup>-1</sup>),  $SO_4^{2-}$  (857 mg  $100^{-1}$  g), organic matter (1.6%), N in all its forms (total-N: 0.15%; nitric-N: 25 mg kg<sup>-1</sup>; soil-N: 94 kg ha<sup>-1</sup>), Zn (0.33 mg kg<sup>-1</sup>), B (3.59 mg kg<sup>-1</sup>), K (1.89 meq  $100^{-1}$  g), Na (6.8 meq  $100^{-1}$ ) and Ca (100.12 meq  $100^{-1}$  g) were significantly higher than in the Villacañas plot (pH:8.2; EC: 0.56 mmhos cm<sup>-1</sup>; C:N ratio: 4.4; Cl<sup>-</sup>: 19.33 mg kg<sup>-1</sup>),  $SO_4$ <sup>=</sup>: 64.3 mg  $100^{-1}$  g; organic matter: 0.7%; total-N: 0.09%; nitric-N: 3.67 mg kg<sup>-1</sup>; soil-N: 14.3 kg ha<sup>-1</sup>; Zn: 0.19 mg kg<sup>-1</sup>; B: 0.12 mg kg<sup>-1</sup>; K: 0.53 meq  $100^{-1}$  g; Na: 0.59 meq  $100^{-1}$  g) and Ca: 25.25 meq  $100^{-1}$  g). On the contrary in Villacañas, the percentages of total  $CO_3$ <sup>2</sup>-(29.45%), the K:Mg (0.56) and Ca:Mg (28.67) ratios, Fe (1.12 mg kg<sup>-1</sup>) and P (9.3 mg kg<sup>-1</sup>) contents were significantly higher than those obtained in Quero ( $CO_3$ <sup>2</sup>-: 14.19%; K:Mg and Ca:Mg ratios: 0.06 and 3.03, respectively; Fe: 0.31 mg kg<sup>-1</sup> and P: < d.l: detection limit).

Regarding SS treatment, no significant differences were observed between Villacañas and Quero with respect to the content of organic matter, total N, total  $CO_3^{2-}$ , Cu, Mn or CEC (**Table 6**).

The differences were significantly greater in Quero regarding pH value (9.01), EC (18.56 mmhos cm $^{-1}$ ), Cl $^{-1}$  (362.6 mg kg $^{-1}$ ), SO $_4$  $^{-1}$  (860 mg 100 $^{-1}$  g), C:N ratio (6.7), nitric N (38 mg kg $^{-1}$ ), soil N (142.6 kg ha $^{-1}$ ), Zn (0.92 mg kg $^{-1}$ ), B (5.8 mg kg $^{-1}$ ), K (2.1 meq 100 $^{-1}$  g), Na (5.8 meq 100 $^{-1}$  g) and Ca (93.28 meq 100 $^{-1}$  g).

		CF				SS			CS		
		P-value	v	Q	P-value	V	Q	P-value	V	Q	
pН		0.0133*	8.2 a	9.14 b	0.0238*	8.4 0a	9.01 b	0.0011*	8.4 a	9.01 b	
EC	mmhos cm <sup>-1</sup>	0.0157*	0.56 a	19.86 b	0.0132*	0.36 a	18.56 b	0.0027*	0.47 a	18.38 b	
Cl <sup>-</sup>	mg kg <sup>-1</sup>	0.0106*	19.33 a	286 b	0.0082*	19.33 a	362.6 b	0.0006*	20.6 a	327.3 b	
SO <sub>4</sub> =	mg 100 <sup>-1</sup> g	0.0000*	64.3 a	857 b	0.0000*	46 a	860 b	0.0000*	42.3 a	860 b	
Total CO <sub>3</sub> =	%	0.0442*	29.45 b	14.19 a	0.1287	39.98 a	15.18 a	0.0386*	32.8 b	13.34 a	
Organic matter	(%)	0.0102*	0.7 a	1.6 b	0.1797	0.99 a	1.86 a	0.053	0.8 a	2.25 a	
Total -N	%	0.0011*	0.09 a	0.15 b	0.1757	0.13 a	0.16 a	0.0035 a	0.103 a	0.200 b	
Nitric-N	mg kg <sup>-1</sup>	0.0020*	3.67 a	25.0 b	0.0016*	8.0 a	38 b	0.0003*	6.3 a	40.6 b	
Soil-N	kg ha <sup>-1</sup> (**)	0.0021*	14.3 a	94.0 b	0.0018*	30.6 a	142.6 b	0.0003*	24.3 a	152.6 b	
Ass. P	mg kg <sup>-1</sup>	0.0030*	9.3 b	<d.1< td=""><td>0.0016*</td><td>12.7 b</td><td><d.1< td=""><td>0.0061</td><td>17.7 b</td><td><d.1< td=""></d.1<></td></d.1<></td></d.1<>	0.0016*	12.7 b	<d.1< td=""><td>0.0061</td><td>17.7 b</td><td><d.1< td=""></d.1<></td></d.1<>	0.0061	17.7 b	<d.1< td=""></d.1<>	
Ass.Fe	mg kg <sup>-1</sup>	0.0183*	1.12 a	0.31 b	0.1177	2.24 a	0.5 a	0.0005*	2.31 b	0.4 a	
Ass.Zn	mg kg <sup>-1</sup>	0.0087*	0.19 a	0.33 b	0.0681	0.38 a	0.92 b	0.2264	0.68 a	0.93 a	
Ass.Cu	mg kg <sup>-1</sup>	0.0948	0.51 a	0.38 a	0.1265	0.62 a	0.47 a	0.4762	0.57 a	0.49 a	
Ass.Mn	mg kg <sup>-1</sup>	0.4285	4.9 a	3.5 a	0.0717	7.3 a	3.8 a	0.0181*	8.7 b	3.9 a	
Ass.B	mg kg <sup>-1</sup>	0.0004*	0.12 a	3.59 b	0.0458*	0.1 a	5.8 b	0.0055*	0.2 a	4.5 b	
CEC	meq 100 <sup>-1</sup> g	0.4398	14.15 a	12.27 a	0.3994	12.9 a	15.2 a	0.1286	13.8 a	15.1 a	
Ass.K	meq 100 <sup>-1</sup> g	0.0048*	0.53 a	1.89 b	0.0023*	0.54 a	2.1 b	0.0005*	0.47 a	2.06 b	
Ass.Na	meq 100 <sup>-1</sup> g	0.0062*	0.59 a	6.8 b	0.0166*	0.65 a	5.8 b	0.0019*	0.67 a	7.17 b	
Ass.Ca	meq 100 <sup>-1</sup> g	0.0001*	25.25 a	100.12 b	0.0001*	26.03 a	93.28 b	0.0000*	24.8 a	100.05 b	

	CF			SS				CS		
	P-value	v	Q	P-value	V	Q	P-value	v	Q	
C:N ratio	0.0438*	4.4 a	6.37 b	0.2076	4.3 a	6.7 b	0.1941	4.5 a	6.5 a	
K:Mg ratio	0.0003*	0.56 b	0.06 a	0.0006*	0.63 b	0.09 a	0.0032*	0.48 b	0.06 a	
Ca:Mg ratio	0.0066*	28.67 b	3.03 a	0.0204*	33.03 b	3.86 a	0.0003*	25.3 b	2.8 a	
* * * * * * * * * * * * * * * * * * * *										

d.l: detection limit.\*Significant differences.

Table 6.

Comparison of the study plot soils (V: Villacañas; Q: Quero) for each fertiliser treatment. Ass.: assimilable. Distinct letters mean groups with significant differences at P < 0.05 between plots for the same treatment according to the LSD test.

In Villacañas, only the values of the K:Mg (0.63) and Ca:Mg (33.03) ratios and Fe (2.24 mg kg<sup>-1</sup>) and P (12.7 mg kg<sup>-1</sup>) content were significantly higher.

Finally, after applying CS, there were no significant differences between Villacañas and Quero for organic matter content, C:N ratio, Zn, Cu and CEC.

The differences in Quero were significantly bigger for pH value (9.01), EC (18.32 mmhos cm<sup>-1</sup>), Cl- (327.3 mg kg<sup>-1</sup>),  $SO_4^{2-}$  (860 mg  $100^{-1}$  g), total N (0.2%), soil N (152.6 kg ha<sup>-1</sup>), B (4.5 mg kg<sup>-1</sup>), K, Na and Ca. In Villacañas, only the values of K: Mg (0.48) and Ca:Mg (25.3) ratios, total  $CO_3^{2-}$  (32.8%), Fe (2.31 mg kg<sup>-1</sup>), P (17.7 mg kg<sup>-1</sup>) and Mn (8.7 mg kg<sup>-1</sup>) contents were significantly higher.

Thus, we described the results according to the applied treatment by comparing the test plots, but by also comparing to the initial soil, how the application of SS, CS and CF affected Villacañas and Quero soils.

Changes noted in the Villacañas soil after harvest in relation to the initial situation when comparing **Tables 3** and **6**. The differences among treatments at the end of the trial are also shown. Regarding the initial soil, no representative changes were observed in pH, EC, nitric N, total  $CO_3^{2-}$ , the K:Mg ratio, assimilable Fe content, Cu, Mn (in SS or CS treatment), CEC and Ca.

Large differences were found in the initial soil for certain parameters. This was the case of the generalised drop in the contents of  $Cl^-$ , organic matter, B, K, Na and the C:N ratio. A reduction was also recorded in the  $SO_4^{2-}$  content of the plots treated with SS and CS, and in assimilable P and Mn in the soils fertilised with CF.

Conversely, the total N and soil N contents in the plots fertilised with SS and CS increased, as did Zn content.

Finally, when comparing treatments, significant differences in the Villacañas plot soil were observed for the total N percentage and assimilable P content. In both cases when SS and CS were applied, the content of these parameters significantly increased compared to the control (CF).

In general, compared to the Villacañas plot, more changes took place in the Quero soil as regards the initial situation after the trial (see **Tables 3** and **6**). Thus, compared to the initial soil, no representative changes were seen in pH, total  $CO_3^{2-}$  or the K:Mg ratio after the applications.

Large differences appeared in the initial soil for most parameters. A generalised decrease in  $SO_4^{2-}$  and assimilable P contents, and in the C:N and Ca:Mg ratios, was observed. A slight decrease in organic matter content was found in the subplots treated with CF and SS.

On the contrary for any of the three treatments, EC, Cl<sup>-</sup> content, total N, nitric N, Mn, B, K, Na, Ca and the CEC all increased compared to the original soil. Soil N, Fe, Zn and Cu in soil also increased, but only with SS and CS.

Finally, when comparing treatments, significant differences were found in the Quero plot soil for the percentage of total N (increased with CS), and for nitric N, N in soil and Zn (increased with SS and CS).

#### 4. Discussion

Soil is where the root system of plants develops, and it provides them with anchorage, water and nutrients. It is also a non-renewable resource on a human time scale that can be lost through erosion or degraded by contamination. Thus, conservation and improvement should be made priorities.

One of the main problems that affects soil productivity is a rising EC by increasing salts [9]. Applying organic amendments, including sewage sludge, has been widely reported to remediate saline soils and to alleviate salinity and sodicity stress in crops [36, 37]. Our study showed that this did not happen in Villacañas, which

coincides with similar studies in which EC did not only not increase, but even lowered [13], which was the case of treatments SS and CS. This could not be stated of the Quero soil. In these subplots, EC increased vs. the original soil, especially with CF. This could be a long-term problem because several crops can have problems developing due to high salinity in soil, which would be a limiting factor for future agricultural uses.

CEC expresses the number of positively charged moles that can be exchanged. A soil's change capacity increases with clay and organic matter content because both have electrical negative charges. The major saturating cations of the change complex are, and in this order: Ca<sup>2+</sup>, Mg<sup>2+</sup> and K<sup>+</sup> (Na appears in alkaline soils) [38]. Compared to the original soils (**Table 3**), CEC changed more in Quero than in Villacañas after treatments, especially with SS and CS (**Table 6**). This may act as an asset to decide about using air-dried sewage sludge or composted sewage sludge for fertilising wheat.

The Ca:Mg ratio rose after treatments (**Tables 3** and **6**), including the control CF in Villacañas, but not in Quero. This means that in Villacañas, Ca content in soil was higher than Mg content, which tends to improve soil aeration. In contrast, together with Zn content could be due to, difficulties in Mg absorption in crops. Dalir et al. [39] reported Zn and Ca and Zn and Ni interactions, as well as high Ca concentrations. This was also the case of the studied Villacañas soils, which caused Zn uptake to drastically drop.

Organic matter in soil plays an essential role in the nutrient cycle [9], which refers to a heterogeneous series of compounds whose origin is biological that are found in different degradation states [38]. As expected, the organic matter content of the tested soils lowered after harvest in all cases, except for treatment CS in the Quero plot. However, the general trend of the studies reviewed in the literature was to use fresh sludge applied to soil as an organic amendment, which increases soil organic matter content [9], P and N [13]. As the literature accurately states, what increased was the total post-harvest nitrogen content with treatments SS and CS, especially in the Quero soil. It is noteworthy that organic matter in organic fertilisers has to be mineralised by soil microorganisms so that nutrients are released to soil. This process is slow and depends on not only organic waste characteristics, but also on soil type and the environment (temperature, soil water content, etc.) [40, 41].

In the Villacañas soil, no post-application changes took place in relation to the initial soil in: pH, EC, nitric N, total carbonates, the K:Mg ratio, content in assimilable iron, copper, manganese (with SS or CS), capacity of cationic exchange and calcium. When comparing treatments, the percentage of total nitrogen and assimilable phosphorus content significantly increased after applying SS and CS versus the control.

In Quero, more changes were observed in relation to the initial soil than in Villacañas. When comparing treatments, a significant increase in the percentage of total N was found for SS and CF, but not for CS, while an increase in CF of nitric N, soil N and Zn content occurred with the application of SS and CS.

The presence of sewage sludge in any studied conformation (SS and CS) promoted higher yields than the commercial fertiliser (CF).

The following were observed in the Villacañas test plot: longer spike length with SS and CS than with CF; heavier average spike weight, average number and weight of grains per spike in the plants fertilised with SS; higher yields for the application of SS, like the findings of Zhang et al. [42]; lower yield with CS.

In the Quero test plot, the following were recorded: higher values for spike length, spike weight, number and weight of grains per spike in all the treatments than their equivalents in Villacañas; longer spike length with SS and CS than with

CF; higher values for spike weight and number of grains per spike with SS; heavier weight of grains per spike with CS application, similarly to other studies [43]; better yields with CS; worse yields with CF.

Like other research works [11, 24], if both air-dried and composted sewage sludge are included in soil fertilisation, the studied crop morphological characteristics improved versus the commercial fertiliser.

In any case, yields were lower in the subplots (CF) than in others (SS and CS), and yield potential correlated positively with the aboveground biomass. Thus, better grain yields were obtained in the crops with a bigger accumulated biomass upon maturity [8]. Our results are consistent with other studies [11], and the good nourishment supported by the high concentration of nutrients in sewage sludge positively influenced the growth of the aboveground biomass of wheat. We verified that, depending on the employed product, differences in yield appeared between the highest and lowest figures, which varied between 10% and 15%. Something similar happened in another study, where the sewage sludge application displayed better fertilising capacity than compost, with 12% bigger wheat yields on average [2]. This response could be associated with different chemical N forms [16]. We cannot rule out the possibility that with the high EC obtained for compost, crops could have difficulties to absorb water because of not being able to overcome osmotic pressure to the consequent detriment of nutrient inputs. Nevertheless, we were able to verify that applying sewage sludge as a soil fertiliser improves both durum wheat yield and productivity and better results than mineral-based fertilisers.

The EU Fertilising Product Regulation (FPR) [44], which comes into force as from 2022, must be considered for a future line of work. Above all, limits for a range of contaminants will be considered, such as Cd, which is contained in mineral fertilisers. With the obtained results, the commercialization of composted sewage sludge would only be possible locally. Its agronomic aptitude has been proven, but the study did not determine standardised production. Future studies will have to carry out composting with a system that better controls parameters than that of controlled piles because this method largely depends on environmental conditions as it is located outdoors. Another possibility to improve this study would be to run tests at different sewage sludge/straw concentrations, and with a different straw size to facilitate mixing.

#### 5. Conclusions

In short, the most important conclusions drawn from our study are:

The effect of two amendments constituted by sewage sludge on soil properties and on wheat (*Triticum aestivum* L.) morphological parameters was evaluated.

Next the application of both air-dried and composted sewage sludge to soil improved the results of the studied wheat morphological characteristics in relation to the commercial fertiliser.

In the Villacañas plot, the best wheat results were obtained by applying air-dried sewage sludge to soil.

On the contrary in the Quero plot, where soil was much more saline and pH was higher, air-dried sewage sludge improved the morphological characteristics of spikes (length, weight and number of grains per spike), but not the final grain weight and, consequently, yields. These were upgraded with composted sewage sludge.

Given the composition of the sewage sludge obtained from the Alcázar de San Juan WWTP, we realized that it was an important source of N and P that serves

to both provide crops with nutrients and to maintain these elements in the soil to which it is applied. Moreover, as it is not ecotoxic waste, it is suitable for agricultural use and might be an adequate substitute for commercial fertiliser. Precisely because of its nitrogen content, it is an excellent raw material for making compost by balancing the C: N ratio together with other structuring agents, such as straw, pruning remains or any other carbon source.

However, the application of this waste is not completely harmless, and several considerations must be considered:

- The influence of soil salinity on the results as blocking effects or synergies of elements that absorb plants can occur. According to our results, it would seem that it is preferable to use saline soil in a composted form.
- Although the applied dose was not herein evaluated, is essential that future field trials learn what the appropriate product quantity is according to soil type and crop type.

Our findings suggest that it is possible to sustain wheat yields and to maintain soil properties when sludge is used as an organic amendment towards more sustainable fertilisation practices.

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