

## Electrical conductivity, pH and other soil chemical parameters after sub-irrigation with untreated and treated municipal wastewater in two different soils

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## Abstract

The aim of this study is to investigate the effect of sub-irrigation with untreated and treated municipal wastewater on soil chemical parameters. Three treatments were used: untreated wastewater (U), treated wastewater (T) and tap water (W), being the control treatment, in two soil types, Sandy loam (SL) and Loamy sand (LS). A subirrigation system including pots filled with soil was installed in one of the greenhouses of the Agricultural University of Athens. The wastewater used was applied in the soil in pots at a depth of 10 cm and 20 cm. In order to determine the variation of chemical parameters at the point where the emitter was placed, the soil was divided into two zones according to depth: (zone I -upper) and (zone II-lower). The pH, CaCO3 %, K μg/g, Na μg/g, P μg/g and EC μmhos/cm, were determined. Statistically significant differences (p < 0.05) in sodium Na<sup>+</sup>  $\mu$ g/g and electrical conductivity EC were observed, only in LS soil. Phosphorus and sodium increased in zone (I) for SL soil (p < 0.05). Electrical conductivity and potassium increased in zone (I), while CaCO<sub>3</sub>% and pH increased in zone (II) for LS soil (P < 0.05).

**Keywords:** Wastewater, soil, sub-irrigation, chemical parameters.

## 1. Introduction

According to UNESCO, fresh water is 2.6 % of the total world water and only a small fraction of its which is in the ground, rivers and the atmosphere can be used for crops irrigation (UNICEF, FAO and SaciWATERs, 2013). The EU Framework Directive for water encourages and promotes treated wastewater in agriculture in order to overcome the problem of water scarcity. The urban wastewater implementation through sub-irrigation could potentially minimize risks to public health, especially for farm workers and consumers of the products of irrigated crops (Forslund *et al.*, 2010). Wastewater sub-irrigation use is considered the best technique to substitute natural water resources and to offer higher returns on crops (Duhrkoop *et al.*, 2014). According to microbiological data given Kiziloglu *et al.* (2008) for a more sustainable agriculture, untreated

wastewater could be used for irrigation but for a short time while treated wastewater could be used for a longer term. Especially in agricultural areas where groundwater has been contaminated, the wastewater application could have a positive effect on soil quality (e.g. organic matter) under right conditions (Hidri et al., 2013). There have been expressed different opinions on the impact of wastewater on soil properties, which may be related to changes in physical, chemical or biological soil properties. In two different soil types, properties were showed important differences as far as conversion of nutrients are concerned, after wastewater application (Magesan et al., 2001). According to Galavi et al. (2010) and Wagner et al. (2006) all soil parameters such as N, P, K, Ca, Na, Mg, SAR, EC, OC % and soil salinity showed a significant increase ( $p \le 0.05$ ) except for pH which decreased after wastewater irrigation. In contrast according to Hidri et al. (2013) pH, organic matter and Cation Exchange Capacity were not affected after drip irrigation with treated wastewater. Munir et al. (2003) observed that not only phosphorus (P), potassium (K), iron (Fe), manganese (Mn) but also Zinc (Zn) and copper Cu) were not significantly affected after irrigation with wastewater. Long-term irrigation with wastewater may create an increase in nutrients to soil (e.g. organic matter, nitrogen (N), salinity, heavy metals and the major of cations concentration) but it may also create a pH reduction (Angin al., 2005); (Bedbabis et et al., 2014). Irrigation with treated wastewater caused a pH decrease but on the other hand it caused a significant increase in organic matter (OM), sodium absorption (SAR) and electrical conductivity (EC) after 4 years. Moreover, electrical conductivity (EC) increased in all types of soil (Bedbabis et al., 2014). According to Jian Xua et al. (2010) a pH reduction was observed in soil depth up to 140 cm after 3, 8 and 20 years of irrigation with wastewater (particularly after 20 years). Furthermore, the total carbon (TOC) content after 80 years of irrigation increased 2.5-fold. Heidarpour et al. (2007) observed that electrical conductivity (EC), sodium (Na<sup>+</sup>) and magnesium (Mg<sup>2+</sup>) increased at the top soil layer (0-15 cm) when

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sub-irrigation was used. Of greatest interest is the EC increase at surface soil layer as it can inhibit growth of plants. Besides, potassium K<sup>+</sup> was higher in upper soil layer at 0-30 cm, while Ca<sup>2+</sup> and Mg<sup>2+</sup> were lower in soil layer of depth of 15-60 cm in both irrigation cases (surface and subsurface). The aim of the present study is to investigate the effect of sub-irrigation with untreated and treated wastewater on electrical conductivity, pH and some other soil parameters in two different soils.

#### 2. Materials and methods

#### 2.1. Experimental description

Experiments were performed in one of the greenhouses of the Agricultural University of Athens. A specific watering system was installed. This system was used for the subsurface application of wastewater into the soil. The soil was packed into pots and sub-irrigation was applied in depths of 10 and 20 cm. Two different soil types were used: SL soil characterized as Sandy Loam and LS soil characterized as Loamy Sand. Three treatments were applied: untreated wastewater (U), treated wastewater (T) and tap water (W) as the control treatment. The wastewater was taken from the biological Wastewater Treatment Plant of Likovrysis in Attica (KEREFYT). At the end of the experimental procedure, soil samples were taken to the laboratory in order to determine their chemical parameters. The following parameters were determined: pH, CaCO3 %, K µg/g, Na µg/g, P µg/g and EC µmhos/cm.

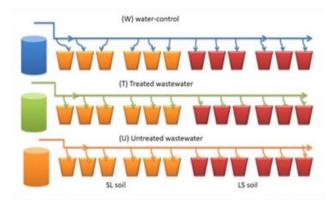


Figure 1. The layout of the pots

### 2.2. Experimental design

For the needs of the experiment, 36 pots were used (Figure 1). The capacity of each pot was 11.8 liters and it was filled with 13 kg of soil. Soil was homogenized, passed through a sieve (having a mesh of 1.0 x 1.0 cm) and was dried in the air (Chen et al., 2000). Then the soil was weighed and was placed into the pots. Each pot contained 13 kg of soil and care was taken in order to ensure equal quantity in all pots. A subsurface drip irrigation system (SDIS) with single drippers was installed for wastewater and/or water application used into to be the pots. The experimental layout consisted of three rows of pots. In the first row of pots, tap water (W) was applied while in the second and the third one treated (T) and untreated wastewater (U) was applied respectively. The tap water

treatment was taken as the control treatment. Each row consisted of 12 pots and three repetitions for each treatment were used. In each pot the above liquids were applied in order to bring the soil at pot capacity ( $\Theta_{FC}$ ) of about 70-75% of the saturated water content. A flow rate of 3 l/h irrigation was applied (Allen et al., 1998). So, each dripper provided 3 l/h of wastewater and/or tap water and the available soil moisture content did not exceed the value of 70-75% of the saturated water content. The system was programmed to operate for 1 hour long, three days a week. For the wastewater application, plastic pipes (diameter 32 and spaghetti type ones (diameter mm) 6 mm) were used. Emitters were connected to spaghetti tubes applying wastewater at a depth of 10 cm and 20 cm below the soil surface. A very fine sieve was used in order not to clog the drippers in the case of untreated wastewater. The LS soil consisted of SL soil mixed with sand at a percentage of 25% in order to make it more permeable to wetting liquids (Tsigoida and Argyrokastritis, 2019). From the 36 pots used, 18 were filled with SL soil and the other 18 were filled with LS soil. Three rows of pots were established and each row consisted of 6 pots with SL soil and another 6 with LS soil. In three of them the emitter was placed at a depth of 10 cm and in the other three ones the emitter was placed at а depth of 20 cm. During the days that irrigation was applied, wastewater was taken in containers to the Agricultural University of Athens. The experiment lasted for six months.

#### 2.3. Soil sampling

At the end of the experimental procedure, soil was removed from each pot so that the shape of the pot is kept. Then, based on the depth (10 and/or 20 cm) at which the emitters were placed, soil was divided into two parts (zones) (Figure 2). The upper part was characterized as zone (I) and the bottom one as zone (II). Soil samples of the two zones were obtained in order to study the variation of soil parameters, in the zones defined by a plane perpendicular to the axis of the pot at the point of injection and compare their values to the ones determined in the control treatment after wastewater addition. Then the samples were taken to the laboratory for soil analysis and the following parameters were determined: pH, CaCO<sub>3</sub> %, K µg/g, Na µg/g, P µg/g and EC µmhos/cm.

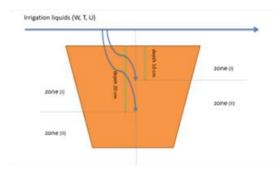


Figure 2. The section of the pot (depths 10, 20 cm)

#### 2.4. Soil chemical analysis

Soil samples were air dried and then passed through a sieve of 2 mm openings. In the soil fraction of particle size < 2

mm, the above mentioned parameters were determined by using the following methods: The soil texture of the soil samples was determined by using the Bouyoukos method (Bouyoukos, 1951). The pH value was determined by using the soil-water suspension at a ratio of 1:1 by using a pHmeter JENWAY 3310. The equivalent calcium carbonate (CaCO<sub>3</sub>) was determined by using the Bernard method (Nelson R.E., 1982). The electrical conductivity was measured at saturation paste by using a Beckman RC1682 meter. The available phosphorus was detected by using the Olsen method (Olsen S.R. and Sommers L.E., 1982).The exchangeable potassium was detected by using the ammonium acetate method CH<sub>3</sub>COONH<sub>4</sub>) (Knudsen D. *et al.*, 1982) and Sodium exchangeable Na<sup>+</sup> by using the same method (Knudsen D. *et al.*, 1982).

## 2.5. Statistical analysis

To assess the differences in the chemical parameters of the soil, the analysis of variance (ANOVA) was used. All statistical analyses were performed at a significance level of  $p \le 0.05$ . When significant effects

were determined ( $p \le 0.05$ ) during multiple comparisons, the Tukey's test was applied in order to find means that are **Table 1.** The wastewater chemical parameters

significantly different from each other. The statistical software package SIGMA STAT was used for all statistical determinations.

#### 2.6. The hydraulic conductivity of soils used

In samples taken from the soils used in the experiments the saturated hydraulic conductivity (Ks), the saturated volumetric water content ( $\Theta$ s) and the soil bulk density ( $\rho_d$ ) were determined and their values are shown as follows: SL soil: *Ks* (*cm*/*h*) = 89.95, *Os* ( $m^3/m^3$ ) = 0.374,  $\rho_d$  ( $t/m^3$ ) = 1.2. LS soil: *Ks* (*cm*/*h*) = 63.19, *Os* ( $m^3/m^3$ ) = 0355,  $\rho_d$  ( $t/m^3$ ) = 1.25.

#### 3. Results and discussion

After 6 months of sub-irrigation with untreated and treated municipal wastewater there were showed some changes in the examined chemical parameters in the soils, which are presented in the figures and tables below. Table 1 shows the mean values of the chemical parameters found in the wastewater used for the experiments before irrigation was applied. As it was expected, the untreated wastewater contained high percentages of SS (mg/l) and COD (mg/l).

| Parameters              | Untreated wastewater (U) | Treated wastewater (T) | Water (W |
|-------------------------|--------------------------|------------------------|----------|
| рН                      | 7.52                     | 7.24                   | -        |
| COD (mg/l)              | 560-988                  | 17.9-23.5              | -        |
| SS (mg/l)               | 235.4                    | 0.71                   | -        |
| NO₃⁻N (µg/ml)           | 0.35                     | 1.48                   | -        |
| NH4 +-N (μg/ml)         | 21.81                    | 0.068                  | -        |
| Total P (μg/ml)         | 4.41                     | 4.08                   | -        |
| K⁺(μg/ml)               | 20                       | 22                     | -        |
| Na + (µg/ml)            | 92                       | 95                     | 70.3     |
| Cl <sup>-</sup> (meq/l) | 0.7                      | 0.6                    | 1.3      |
| EC (µS/cm)              | 1090                     | 814                    | 920      |
| T H (meq/l)             | 4.3                      | 4.1                    | 6.75     |
| SAR                     | 2.73                     | 2.88                   | 1.66     |

#### 3.1. The chemical parameters determined for SL and LS soils

In Table 2 the average values and the standard deviation in the chemical analysis results for SL and LS soils are presented, while in Table 3 one way analysis of variance (ANOVA) results for the same parameters and for the same soils are shown respectively. In Figure 3 we can see the differences in pH, CaCO3 %, K  $\mu$ g/g, Na  $\mu$ g/g, P  $\mu$ g/g and in EC  $\mu$ mhos/cm between SL and LS soil at the end of the irrigation period with the three wetting liquids (W, T, U). Differences in pH, CaCO3 %, K  $\mu$ g/g, Na  $\mu$ g/g, P  $\mu$ g/g and EC  $\mu$ mhos/cm of SL and LS soils are found as follows.

## 3.1.1. pH: (Figure 3)

At the end of the irrigation period with treated T and untreated U wastewater in SL and LS soils, there was a slight decrease in treatment with untreated U but a slight increase with treated T compared to the control (W) treatment. Among the treatments in two soils, there were no statistically significant differences (p> 0.05) (see Table 4). Comparison between the three treatments as well as comparison between the two soils showed no statistically significant differences p>0.005 (see Tables 5 and 6). Besides, there was no difference for the same treatment between the soils SL and LS (see Table 7). Chahal *et al.* (2011); Qian and Mecham (2005); Mohammad and Mazahreh (2003); Hidri *et al.* (2013 reached the same conclusion.

3.1.2. The equivalent calcium carbonate CaCO<sub>3</sub>%: (Figure 3) It was found that a slight decrease and an increase in both treatments (T, U) in SL soil and in LS soil were showed respectively, compared to the control treatment (W). Among the treatments in two soils there were no statistically significant differences (p> 0.05) (see Table 4). Regarding the comparison between treatments, there were showed statistically significant differences between U vs W (p>0.005) (see Table 5). Similarly, statistically significant differences between the two soils SL and LS (p < 0.001) were observed (see Table 6). Moerover, a statistically significant difference was found in the same treatment (W<sub>a</sub> Vs W<sub>β</sub>) for soils SL and LS (see Table 7). Qian and Mecham (2005) however, observed a small reduction in CaCO3% when soil irrigated with treated wastewater. Chahal *et al.* (2011); Abegunrin (2016); Schipper *et al.* (1996) used soil columns and they found that Ca<sup>++</sup> and Mg<sup>++</sup> concentrations are greater in ablutions than in applied wastewater, indicating that added cations (Ca<sup>++</sup> and Mg<sup>++</sup>) with wastewater leached through soil. Furthermore, Chahal *et al.* (2011) found that the cation concentrations (Ca<sup>++</sup> and Mg<sup>++</sup>) were significantly (p < 0.05) higher in treated wastewater than in the control treatment in soil columns. According to Everett M., *et al.*(2007) these cations (Ca<sup>++</sup> and Mg<sup>++</sup>) concentrations did not change significantly but there were observed some chages in soil physical characteristics. According to Rattan *et al.* (2005) irrigation with treated wastewater causes a redistribution of salts in the soil profile and also in some elements which have the tendency to accumulate in the soil surface. Lado *et al.* (2012) noted that in sandy soil (noncalcareous sandy) and clay soil (calcareous clayey) the salts increased in the upper layer (1 m) after wastewater irrigation but these salts are washed during precipitation below a depth of 1.5 m.

| Parameters            | Samples | oles Untreated wastewater U |       | Treated wastewater T |       |        | Control W |       |       |       |       |       |       |
|-----------------------|---------|-----------------------------|-------|----------------------|-------|--------|-----------|-------|-------|-------|-------|-------|-------|
|                       | No      | SL                          | soil  | LS s                 | oil   | SL s   | oil       | LS    | soil  | SL    | soil  | LS    | soil  |
|                       |         | avg                         | Sd    | avg                  | Sd    | avg    | Sd        | avg   | Sd    | avg   | Sd    | avg   | Sd    |
| рН                    | 72      | 8.24                        | 0.21  | 8.19                 | 0.19  | 8.37   | 0.27      | 8.41  | 0.23  | 8.34  | 0.28  | 8.31  | 0.25  |
| Equivalent CaCO₃<br>% | 72      | 26.14                       | 1.57  | 25.66                | 2.40  | 26.29  | 1.69      | 25.11 | 1.32  | 27.06 | 2.02  | 23.92 | 1.60  |
| K μg/g                | 72      | 55.23                       | 4.20  | 55.5                 | 3.16  | 56.33  | 13.95     | 53.37 | 8.18  | 55.5  | 5.19  | 57.81 | 9.85  |
| Na µg/g               | 72      | 98.76                       | 18.93 | 84                   | 4.89  | 102.3  | 41.41     | 74.87 | 12.41 | 78.83 | 26.83 | 52.90 | 21.45 |
| P μg/g                | 72      | 6.99                        | 2.64  | 5.80                 | 2.62  | 6.75   | 2.77      | 6.58  | 1.00  | 6.21  | 3.43  | 5.27  | 2.49  |
| EC µmhos/cm           | 72      | 2050                        | 674.5 | 1527.5               | 244.4 | 2291.6 | 1249.     | 1567  | 400.8 | 1478  | 366.1 | 1029  | 132.7 |

Table 3. One way analysis of variance (ANOVA) in each chemical parameter for SL and LS soils

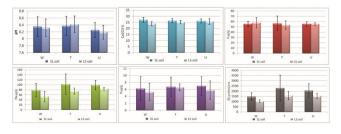
| Parameters                     | SL     | SL soil |         | soil   | - Tast. One way analysis of variance (ANOVA) $n < 0.01$                    |  |
|--------------------------------|--------|---------|---------|--------|--|--|
| Parameters                     | avg    | Sd      | avg     | Sd     | <ul> <li>Test: One way analysis of variance (ANOVA) p &lt; 0.05</li> </ul> |  |
| рН                             | 8.34   | 0.28    | 8.31    | 0.25   | Normality failed, K-W analysis p = 0.995                                   |  |
| Equivalent CaCO <sub>3</sub> % | 27.06  | 2.02    | 23.92   | 1.60   | Normality failed, K-W analysis (Tukey) p = 0.004 (SS)                      |  |
| K⁺ µg/g                        | 55.5   | 5.19    | 57.81   | 9.85   | Normality failed, K-W analysis p = 0.495                                   |  |
| Na⁺ µg/g                       | 78.83  | 26.83   | 52.90   | 21.45  | Normality failed, K-W analysis (Tukey) p = 0.002 (SS)                      |  |
| P μg/g                         | 6.21   | 3.43    | 5.27    | 2.49   | p = 0.826  |  |
| EC µmhoS/cm                    | 1477.5 | 366.16  | 1029.09 | 132.77 | Equal variance failed, K-W analysis (Tukey) p = 0.001 (SS)                 |  |

Note: K-W analysis = Kruskal-Wallis one way analysis on ranks, SS: statistically significant

#### 3.1.3. Potassium: (Figure 3)

In the two treatments (T, U) no difference was detected for SL soil while for LS soil a slight decrease in both treatments (T, U) compared to the control treatment was found, which was not statistically significant. Among the treatments, there were no statistically significant differences for SL and LS soils (see Table 4). Comparison between treatments showed no statistically significant differences (p>0.05) (see Table 5). Also between the soils SL and LS (see Table 6) and between the same treatments in the two soils no statistically significant differences were reported (see Table 7). Nyamangara and Mzezewa (2000); Qian and Mecham (2005) in their experiments found that K<sup>+</sup> concentration decreased by depth. On the other hand, Abegunrin (2016); Monnett et al. (1996); Fuentes et al. (2002) noted that potassium increased during irrigation period with wastewater, which accounts for potassium increase in soil. Likewise, in other studies such as Heidarpour et al. (2007); Wagner Walker de Alb, et al., 2006; Galavi et al., 2010) it was reported that potassium increased in soil and according to Mohammad and Mazahreh (2003) this increase was attributed to high content of the wastewater applied. Rusan et al. (2007) observed that potassium remained for a long time in soil even after the end of wastewater application. Potassium concentrations were lower in outflow than inflow, showing retention in soil profile when wastewater was applied in soil columns (Chahal *et al.*, 2011). Furthermore, potassium increased in forage plants more than what it was necessary after irrigation with wastewater (Rusan *et al.*, 2007). It is worth mentioning that nitrate, phosphorus and potassium concentrations are much lower in untreated than in treated wastewater (Yadav *et al.*2002).

The differences in chemical parameters between SL and LS soils are shown in the following figures.



**Figure 3.** Differences in a) pH, b) CaCO3 %, c) K  $\mu$ g/g, d) Na  $\mu$ g/g, e) P  $\mu$ g/g and f) EC  $\mu$ mhos/cm of SL and LS soils, at the end of the irrigation period with three wetting liquids (W, T, U). The vertical bars represent the standard deviation of the mean values (n = 3)

#### ELECTRICAL CONDUCTIVITY, PH AND OTHER SOIL CHEMICAL PARAMETERS AFTER SUB-IRRIGATION

| Parameters -                   |       | One way analysis of variance-ANOVA (U vs T vs W) p < 0.05 |  |  |  |
|--------------------------------|-------|---|--|--|--|
|                                |       | SL soil   | LS soil  |  |  |
| рН                             | W-T-U | P = 0.590   | P = 310  |  |  |
| Equivalent CaCO <sub>3</sub> % | U-T-W | P = 0.116   | K-W analysis p = 0.061   |  |  |
| K⁺ μg/g                        | W-T-U | (normality failed) K-W analysis p = 0.988                 | (normality failed) K-W analysis p = 0.163                                |  |  |
| Na⁺ µg/g                       | W-T-U | P = 0.376   | Tukey test p = < 0.001, normality passed equal Variance test passed (SS) |  |  |
| P μg/g                         |       | P = 0.530   | normality failed, K-W analysis p = 0.711                                 |  |  |
| EC μmhos/cm                    | W-T-U | (normality failed) K-W analysis p = 0.065                 | Normality passed equal Variance test passed, Tukey test p = < 0.001 (SS) |  |  |

## Table 4. One way analysis of variance (ANOVA) among three treatments (W, T, and U) for SL and LS soil

Note: K-W analysis = Kruskal-Wallis one way analysis on ranks, SS: statistically significant

## Table 5. t-test analysis between two treatments for each chemical parameter for SL soil and for LS soil

|                                |                    |                    | Test: t-test          | p < 0.05                  |                         |                    |  |
|--------------------------------|--------------------|--------------------|-----------------------|---------------------------|-------------------------|--------------------|--|
| Deverseters                    |                    | SL soil            |                       | LS soil                   |                         |                    |  |
| Parameters                     | U vs W             | T vs W             | T vs U                | U vs W                    | T vs W                  | T vs U             |  |
| pН                             | P = 0.556          | p = 0.680          | p = 0.288             | M-W test p = 0.338        | p = 0.410               | p = 0.136          |  |
| Equivalent CaCO <sub>3</sub> % | P = 0.051          | p = 0.181          | p = 0.528             | M-W test p = 0.0.033 (SS) | M-W test p = 0.105      | p = 0.562          |  |
| K μg/g                         | M-W test p = 0.837 | M-W test p = 0.861 | M-W test p =<br>0.794 | p = 0.328                 | p = 0.161               | M-W test p = 0.226 |  |
| Na µg/g                        | p = 0.204          | p = 0.239          | M-W test p =<br>0.418 | p < 0.001 (SS)            | M-W test p = 0.003 (SS) | p < 0.011 (SS)     |  |
| P μg/g                         | p = 0.284          | p = 0.548          | p = 0.595             | p = 0.412                 | M-W test p = 0.312      | M-W test p = 1.000 |  |
| EC µmhos/cm                    | p = 0.024 (SS)     | M-W test p = 0.060 | p = 0.977             | M-W test p < 0.001 (SS)   | M-W test p < 0.001 (SS) | p = 0.048 (SS)     |  |

*Note: M*-*W* test = *Mann*-*Whitney Rank Sum* test, **SS**: statistically significant

## 3.1.4. Sodium: (Figure 3)

Both SL and LS soils in two treatments (T, U) showed a considerable increase compared to the control treatment (W), which is greater in T treatment. The comparison among the treatments, the comparison between two treatments in each soil as well as the comparison between SL and LS soils all showed statistically significant differences p < 0.005. (see Tables 4–6). Besides, there were found differences for the same treatment between the soils SL and LS (see Table 7). Galavi et al. (2010) and Qian and Mecham (2005) used treated wastewater for irrigation and they observed a significant increase in Na<sup>+</sup> which affected the soil physical characteristics and they noted that even at a slight increase of sodium these characteristics changed (Everett et al., 2007). Furthermore, long-term irrigation with wastewater increased Na<sup>+</sup> content which is attributed to the high basic cations contained in the wastewater, such as Na<sup>+</sup>, Ca<sup>++</sup> and Mg<sup>++</sup> (Schippe et al., 1996). Other studies showed that sodium adsorption ratio (SAR) and sodium and chlorine level increased significantly when treated wastewater was used for irrigation (Wagner Walker de Alb. et al., 2006); (Hentati et al., 2013) but according to Abegunrin et al. (2016) this was an increase in the soil top layer of depth of 0-20 cm. In other studies, in the sodium absorption ratio (SAR) there were no significant differences and if there was any, still the water which was used for irrigation contained a high concentration of sodium (Alrajhi et al., 2015). Sodium absorption ratio (SAR) increased in a depth of at least 4.0 meters in sandy soils (noncalcareous sandy soil) and less (0.7 meters) in clayey soils (calcareous clayey soil), when they were irrigated with treated wastewater (Lado et al., 2012). Sodium concentration was detected at different depths (up to 120 cm), when soil was irrigated for more than 10 years with treated wastewater (Levy, Guy et al., 2014). In soil solution, between the ESP (Exchangeable Sodium Percentage) and SAR (Sodium Adsorption Ratio) there is a balance. This balance indicates that there is a chemical equilibrium in the exchanging process between soil and soil solution. So it is further concluded that the properties in soil solution are not always dictated by irrigation water (Levy, Guy et al., 2014). Halliwell (2001) reported that Exchangeable Sodium Percentage (ESP) values caused some changes in soil sodicity. These changes may be due to the soil different minerals and lack of the electrolyte concentrations in the soil solution, which caused small ranges in (ESP) values (Halliwell et al., 2001). In some studies such as Rattan et al. (2005); Herpin et al. (2007); Page et al. (1986); Adhikari et al. (2014) it was found that irrigation with treated wastewater caused a redistribution of salts in the soil profile and an accumulation trend of some elements in the soil surface. Furthermore, Na<sup>+</sup> concentrations were

lower in outflow than in inflow in soil columns when wastewater was applied, showing their retention in soil profile (Chahal *et al.*, 2012). In other studies there was no significant effect on soil Na<sup>+</sup> concentration after irrigation with wastewater. Na<sup>+</sup> concentration was affected by the water movement in soil, by sodium content in the irrigation water and even by its uptake by plants (Heidarpour *et al.*, 2007).

## 3.1.5. Phosphorus: (Figure 3)

As it can be seen in SL and LS soils in both treatments with (T, U) there was an increase. More specifically, the increase was greater in T treatment in SL soil but in LS soil it was greater in U treatment, compared to the control treatment (W) not statistically significant differences (p> 0.05), though. However, in all other comparisons there were not observed any statistically significant differences (p> 0.05) (see Tables 4–7). According to Yadav et al. (2002), phosphorus concentration is significantly lower in untreated than in treated wastewater. Studies in outdoor experiments (Rusan et al., 2007; Qian and Mecham., 2005) concluded that phosphorus concentration increased in soil during irrigation with treated wastewater. Moreover, in other studies it was reported that phosphorus increased as the duration of irrigation with wastewater continued (Monnett et al., 1996) (Fuentes et al., 2002) (Belaid et al., 2012) (Cooper et al., 20015). In fact, phosphorus increased up to 4, 8 and 10 times in some cases, depending on time of irrigation with wastewater (Rusan et al., 2007). Therefore, it seems that phosphorus contained in wastewater is transported in soil (Munir et al., 2003; Heidarpour et al., 2007). Besides that, phosphorus concentration in soil is influenced by water movement and by its concentration in the irrigation water and even by plants uptake (Heidarpour et al., 2007). On the whole, total % of phosphorus increased in soil after irrigation with treated wastewater. Therefore, PO<sub>4</sub>-P showed a significant reduction in filtered water of an unsaturated sand layer, when secondary treatment wastewater was applied (Bali et al., 2011).Regarding soil columns, PO<sub>4</sub> concentration was lower in the effluent than in the applied wastewater contained, indicating its retention in soil profile (Chahal et al., 2011). Moreover, phosphorus was reduced by 30% in a thick limestone layer (vadose zone-9 m) when secondary treated wastewater penetrated it (Bekele et al., 2011) and it was removed by 94% (53 tones) in an area (100 hectares) irrigated with wastewater (Kadlec, 2009). About half of the total PO<sub>4</sub>-P was absorbed and turned into calcium phosphate in the soil upper laver (91 m), when treated wastewater was applied into poorly drained soil for over 4 years (Everett et al., 2007).

Table 6. One way analysis of variance (ANOVA) between soils SL and LS

| Parameters                     | Test: one way analysis of variance-ANOVA (SL soil Vs LS soil)<br>Normality passed, P = 0.811 |  |
|--------------------------------|--|--|
| рН                             |  |  |
| Equivalent CaCO <sub>3</sub> % | Normality passed, Holm-Sidak method: P < 0.001 (SS)  |  |
| K⁺ µg/g                        | Normality failed, K-W analysis P = 0.798   |  |
| Na⁺µg/g                        | Normality passed, Holm-Sidak method: P < 0.001 (SS)  |  |
| P μg/g                         | Normality passed, P = 0.261  |  |
| EC μmhos/cm                    | Normality failed, Dunn's method: P < 0.001 (SS)  |  |

Note: SS: statistically significant

Table 7. One way analysis of variance (ANOVA) for the same treatment (W, T and U) between the soils SL and LS

| Parameters                     |                             | Test: one way analysis of variance-ANOVA (SL soil Vs LS soil) |
|--------------------------------|-----------------------------|---|
| _                              | $W_{\alpha}$ Vs $W_{\beta}$ | Normality passed, P = 0.976                                   |
| рН                             | $T_{\alpha}$ Vs $T_{\beta}$ | Normality failed, K-W analysis P = 0.761                      |
| _                              | $U_{\alpha}$ Vs $U_{\beta}$ | Normality passed, P = 0.983                                   |
| _                              | $W_{\alpha}$ Vs $W_{\beta}$ | Normality failed, Tukey P = 0.004 (SS)                        |
| Equivalent CaCO <sub>3</sub> % | $T_{\alpha}$ Vs $T_{\beta}$ | Normality passed, P = 0.086                                   |
|                                | $U_{\alpha}$ Vs $U_{\beta}$ | Normality failed, K-W analysis P = 0.877                      |
|                                | $W_{\alpha}$ Vs $W_{\beta}$ | Normality passed, P = 0.265                                   |
| K⁺ µg/g                        | $T_{\alpha}$ Vs $T_{\beta}$ | Normality failed, K-W analysis P = 0.523                      |
|                                | $U_{\alpha}$ Vs $U_{\beta}$ | Normality passed, P = 0.681                                   |
| _                              | $W_{\alpha} Vs W_{\beta}$   | Holm Sidak, Normality passed, P = 0.002(SS)                   |
| Na⁺ µg/g                       | $T_{\alpha}$ Vs $T_{\beta}$ | Normality failed, Tukey, K-W analysis P = 0.019 (SS)          |
|                                | $U_{\alpha}$ Vs $U_{\beta}$ | Holm Sidak, Normality passed, P = 0.030 (SS)                  |
| _                              | $W_{\alpha} Vs W_{\beta}$   | Normality passed, P = 0.620                                   |
| P μg/g                         | $T_{\alpha}$ Vs $T_{\beta}$ | Normality passed, P = 0.727                                   |
|                                | $U_{\alpha}$ Vs $U_{\beta}$ | Normality passed, P = 0.248                                   |
|                                | $W_{\alpha} Vs W_{\beta}$   | Equal Variance failed, Tukey, K-W analysis $P \le 0.001(SS)$  |
| EC µmhos/cm                    | $T_{\alpha}$ Vs $T_{\beta}$ | Normality failed, K-W analysis P = 0.148                      |
|                                | $U_{\alpha}$ Vs $U_{\beta}$ | Holm Sidak, Normality passed, P = 0.011 (SS)                  |

Note:  $(W_{\alpha}, W_{\beta}), (T_{\alpha}, T_{\beta}), (U_{\alpha}, U_{\beta}): \alpha = SL$  soil and  $\beta = LS$  soils n three treatments (water, treated wastewater, untreated wastewater), **SS**: statistically significant

#### 3.1.6. Electrical conductivity (EC)

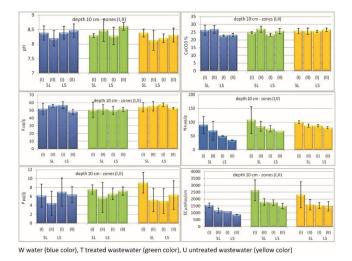
Electrical conductivity (Figure 3) showed an increase in both treatments (T, U) compared to the control (W) treatment, in SL and LS soils with a larger increase being observed in T treatment. Still, the increase was not statistically significant for SL soil but statistically significant for LS soil. Among the treatments, there were statistically significant differences (p < 0.001) in LS soil (see Table 4). The comparison between treatments showed statistically significant differences in SL soil between U and W and between all treatments in LS soil (p < 0.05) (see Table 5). The comparison between the two soils also showed significant differences (p < 0.05) (see Table 6). Besides, there was a difference for the same treatment between the soils SL and LS (p < 0.05) except for  $T_{\alpha}$  Vs  $T_{\beta}$  (see Table 7). Bedbabis et al. (2014); Wagner Walker de Alb. et al. (2008); Gross et al. (2005); Qian and Mecham (2005) also found that electrical conductivity (EC) increased, when treated wastewater was used for irrigation. According to Chahal et al. (2011) the soil EC increase can be due to the wastewater used for irrigation. Galavi et al. (2010) also found that a soil EC increase between 2.9 to 4.52 (DS m-1) after wastewater irrigation. Furthermore, Bedbabis et al. (2014); Galavi et al. (2010) reported an increase in SAR ( $p \le 0.05$ ) whereas Rusan et al. (2007) noted an increase in soil salinity and in soluble salts.

Moreover, Siebe and Cifuentes (1995) and Tabari and Salehi (2008) observed an increase in heavy metals and in soil surfactants concentration, when a soil was irrigated with wastewater. Mohammad and Mazahreh (2003) concluded that this increase can be attributed to the initial high load of total dissolved solids (TDS) which contained in wastewater. The soil EC increased after some years (3, 8 and 20 years) of irrigation with wastewater (Xua Jian et al., 2010). It was reported that soil EC increased by the presence of soluble salts (sodium, magnesium and calcium) contained in the wastewater during irrigation period (Fuentes et al., 2002). Furthermore, high EC values and high Na<sup>+</sup> and Ca<sup>2+</sup> concentrations contained in treated wastewater led to SAR increased values and to the exchangeable Na<sup>+</sup> and Ca<sup>2+</sup> in soil after irrigation (Page et al., 1998). In calcareous soil extracts, EC is very high at all depths (up to 4 mS/cm,) due to the significant cations supply even in the deepest layers to which sometimes EC is greater (Belaid et al., 2012). Between inflow and outflow of soil columns to which wastewater was applied, the EC values showed to be at the same level (Lian et al., 2013). Finally, hydraulic properties did not show any significant changes when in clay soil irrigated with treated wastewater (Cirelli et al., 2012).

3.2. The variation of the chemical parameters between zones (I) and (II)

#### 3.2.1. pH: (Figures 4 and 5)

It can be seen that pH in two zones (I) and (II) showed a small decrease in treatment with untreated (U) in zone (I) in the depth of 20 cm in LS soil (statistically significant (p = 0.003) (see Table 9). According to Hidri *et al.* (2013); Mohamed and Mazahreh (2003); Walker and lin (2007); Chahal *et al.* (2011); Belaid *et al.* (2012) there was a significant decrease in pH after irrigation with treated wastewater as well as in calcareous soil (Bedbabis *et al.*, 2014) particularly in the soil upper layer (0-30 cm) (Kiziloglu, *et al.*, 2008).



**Figure 4.** The variation of a) pH, b) CaCO3 %, c) K  $\mu$ g/g, d) Na  $\mu$ g/g, e) P  $\mu$ g/g and f) EC  $\mu$ mhos/cm in two zones (I) and (II) at a depth of 10 cm in SL and LS soils at the end of the irrigation period with three wetting liquids (W, T, U). The vertical bars represent the standard deviation of the mean values (n = 3)

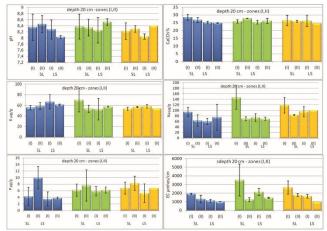
# 3.2.2. The equivalent calcium carbonate CaCO<sub>3</sub>%: (Figures 4 and 5)

CaCO<sub>3</sub>% showed an increase in the control treatment (W) at both depths in zone (II) for SL soil and in treatment with untreated wastewater (20 cm depth) (statistically significant p < 0.05) for LS soil (see Tables 8 and 9). According to Abu-Awwad. (1996) calcium salts accumulate in deeper soil layers due to leaching from the surface area. Heidarpour *et al.* (2007) marked an increase of Ca<sup>++</sup> and Mg<sup>++</sup> in soil zone at a depth of 15-30 cm whereas Majed P.*et al.* (1999); Kiziloglu, *et al.* (2008) found an increase of CaCO<sub>3</sub>% in the zone of a depth 30-60 cm and in the zone of a depth 0-30 cm in calcareous soil respectively, after irrigation with treated wastewater.

## 3.2.3. Potassium: (Figures 4 and 5)

We can see that in SL no changes were observed in potassium. For LS soil an increase in treatment (U) in zone (I) was observed (statistically significant p = 0.035 (p < 0.05) as well as in the control treatment p = 0.047 (p < 0.05) (see Table 8). According to Rusan *et al.* (2007), K<sup>+</sup> was increased as the wastewater application continued in the upper soil layer (0-20 cm). This is supported by other studies in which the amount of K<sup>+</sup> was significantly larger in

the first and in the second soil layer (0-15 cm and 15-30 cm), when it was irrigated with wastewater (Heidarpour et al., 2007), even at a depth of 50 cm (Majed P. et al., 1999). Moreover, in calcareous soil the exchangeable K<sup>+</sup> increased particularly in the soil layer (0-30 cm) (Kiziloglu, et al., 2008). On the other hand, K<sup>+</sup> increased in all layers in a calcium soil which was irrigated with treated wastewater. This increase can be attributed to the relatively high concentration of this cation contained in the treated wastewater. This increase can be attributed to the relatively high concentration of this cation contained in the treated wastewater. This cation can be enhanced by calcium carbonate in the deepest layer (Belaid N. et al., 2012) In a clay soil, K<sup>+</sup> concentration increased in soil layer above the drip lines, after sub-irrigation with wastewater (Jiajie Hea et al., 2013). According to Belaid N. et al. (2012), no changes were observed for K<sup>+</sup> in zones at a depth of 0-30 cm and 30-50 cm during irrigation with wastewater.



**Figure 5.** The variation of a) pH, b) CaCO3 %, c) K  $\mu$ g/g, d) Na  $\mu$ g/g, e) P  $\mu$ g/g and f) EC  $\mu$ mhos/cm in two zones (I) and (II) at a depth of 20 cm in SL and LS soils at the end of the irrigation period with three wetting liquids (W, T, U). The vertical bars represent the standard deviation of the mean values (n = 3) 2.2.4. Codium (figure 4 and 5)

## 3.2.4. Sodium: (Figures 4 and 5)

In SL soil, a reduction in sodium in zone (II) was observed in T treatment at the depth of 20 cm, which was statistically significant p = 0.037 (p < 0.05). However, in LS soil a reduction was observed in zone (II) in the control treatment, which was also statistically significant p = 0.019 (p < 0.05) (see Tables 8 and 9) but this was not the case in the other treatments. Belaid et al. (2012) showed that Na<sup>+</sup> increased in soil layers of 0-10 cm and of 10-30 cm compared to 30-50 cm layer, because of sodium supply contained in the treated water which was used. According to Majed P. et al. (1999), Na<sup>+</sup> changed at various soil depths (0-25 cm, 25-50 cm, 50-100 cm, 100-150 cm and 150-200 cm) after irrigation with treated wastewater and it was also shown that Na<sup>+</sup> content increased at all depths other than 100-150 cm depth. Furthermore, Heidarpour et al. (2007) found that Na<sup>+</sup> and Mg<sup>2+</sup> were significantly greater in the first soil zone (0-15 cm) after sub-irrigation with wastewater compared to surface irrigation. Even in a calcareous soil, which was irrigated with treated wastewater, it was found that exchangeable Na<sup>+</sup> increased especially in soil layer 0-30 cm (Kiziloglu et al., 2008). According to other researchers the exchangeable Mg<sup>2+</sup>and

Na<sup>+</sup> content increased in all soil layers in a calcareous soil (Belaid *et al.*, 2012). This increase was significant in the upper soil layer and this can be attributed to their high concentrations in treated wastewater, while it can be enhanced by wastewater washing through the calcium carbonate in the deepest layers (Belaid *et al.*, 2012). In a clay soil, Na<sup>+</sup> concentration increased in the soil layer above the drip lines after sub-irrigation with wastewater (Jiajie Hea *et al.*, 2013).

## 3.2.5. Phosphorus: (Figures 4 and 5)

In SL soil an increase was found in U treatment in zone (I) which was statistically significant (p = 0.037) (p < 0.05). Besides, there was an increase in the control (W) treatment in zone (I) at a depth of 10 cm and in zone (II) at a depth of 20 cm, statistically significant (p < 0.05)

(p = 0.002 and p = 0.008) respectively (see Tables 8 and 9). In LS soil there were not observed any statistically significant differences in phosphorus. Rusan *et al.* (2007) reported a phosphorus increase in the soil first layer (0-20 cm) after wastewater irrigation for a long time and according to Majed P. *et al.* (1999) phosphorus increased markedly in the upper soil layer 0-25 cm compared to depths 25-50 cm, 50-100 cm, 100-150 cm and 150-200 cm.

Moreover, Lado *et al.* (2012) observed that phosphorus concentrated at the top of the soil profile (100 cm) after irrigation with wastewater. It is also worth mentioning that phosphorus increased by about 80% in the soil upper layer compared to subsoil (Belaid N. *et al.*, 2012), while the available phosphorus increased in a limestone soil especially at a of layer 0-30 cm (Kiziloglu *et al.*,2008).

Table 8. t-test analysis between the chemical parameters in zones (I) and (II) for SL and LS soils at 10 cm depth

| Parameters                     |             | Test: t-test             | p < 0.05                                  |
|--------------------------------|-------------|--------------------------|---|
| Parameters                     | Depth 10 cm | SL soil                  | LS soil                                   |
|                                | W (I)-(II)  | 0.017 M-W test p = 0.100 | 0.702 normality failed M-W test p = 1.000 |
| рН                             | т (I)-(II)  | 0.461                    | 0.157                                     |
|                                | U (I)-(II)  | 0.157                    | 0.529                                     |
|                                | W (I)-(II)  | 0.026 (SS)               | 0.943                                     |
| Equivalent CaCO <sub>3</sub> % | T (I)-(II)  | 0.392                    | 0.026 (SS)                                |
|                                | U (I)-(II)  | 0.238                    | 0.602                                     |
|                                | W (I)-(II)  | 0.121                    | 0.047 (SS)                                |
| K⁺ µg/g                        | т (I)-(II)  | 0.926                    | 0.238                                     |
|                                | U (I)-(II)  | 0.801                    | 0.035 (SS)                                |
|                                | W (I)-(II)  | 0.251                    | 0.019 (SS)                                |
| Na⁺ µg/g                       | т (I)-(II)  | 0.492                    | 0.281 normality failed M-W test p = 1.000 |
|                                | U (I)-(II)  | 0.450                    | 0.093                                     |
|                                | W (I)-(II)  | 0.002 M-W test p = 0.100 | 0.921                                     |
| P μg/g                         | т (I)-(II)  | 0.423 M-W test p = 0.700 | 0.541                                     |
|                                | U (I)-(II)  | 0.037 (SS)               | 0.608                                     |
|                                | W (I)-(II)  | 0.519                    | < 0.001 (SS)                              |
| EC µmhos/cm                    | T (I)-(II)  | 0.138                    | 0.224                                     |
|                                | U (I)-(II)  | 0.168                    | 0.808                                     |

Note: M-W test = Mann-Whitney Rank Sum test, **SS**: statistically significant

Table 9. t-test analysis between the chemical parameters in zones (I) and (II) for SL and LS soils at 20 cm depth

| Daramators                     |             | Test: t-test p < 0                        | .05  |
|--------------------------------|-------------|---|--|
| Parameters                     | Depth 20 cm | SL soil                                   | LS soil  |
|                                | W (I)-(II)  | 0.072                                     | 0.147  |
| pН                             | ⊤ (I)-(II)  | 0.894                                     | 0.337  |
|                                | U (I)-(II)  | 0.698                                     | 0.003 (SS)                                     |
|                                | W (I)-(II)  | 0.039 (SS)                                | 0.710  |
| Equivalent CaCO <sub>3</sub> % | T (I)-(II)  | 0.154                                     | 0.513  |
|                                | U (I)-(II)  | 0.567                                     | 0.830 equal variance failed M-W test p = 1.000 |
|                                | W (I)-(II)  | 0.158                                     | 0.359  |
| K⁺ µg/g                        | ⊤ (I)-(II)  | 0.289                                     | 0.710  |
|                                | U (I)-(II)  | 0.152                                     | 0.596  |
|                                | W (I)-(II)  | 0.006 (SS)                                | 0.535  |
| Na⁺ µg/g                       | T (I)-(II)  | 0.037 normality failed M-W test p = 0.100 | 0.625  |
|                                | U (I)-(II)  | 0.095                                     | 0.681  |
|                                | W (I)-(II)  | 0.008 (SS)                                | 0.458  |
| P μg/g                         | T (I)-(II)  | 0.658                                     | 0.715  |
|                                | U (I)-(II)  | 0.421                                     | 0.676 equal variance M-W test p = 1.00         |
|                                | W (I)-(II)  | 0.618                                     | 0.880  |
| EC µmhos/cm                    | ⊤ (I)-(II)  | 0.101                                     | 0.054  |
|                                | U (I)-(II)  | 0.124                                     | 0.263  |

Note: M-W test = Mann-Whitney Rank Sum test, SS: statistically significant

#### 3.2.6. Electrical conductivity (EC): (Figures 4 and 5)

It can be seen that EC showed a decrease in zone (II) at the two depths (statistically significant (p < 0.05)) in SL and LS soils (see Tables 8 and 9). Heidarpour et al. (2007) and Page et al. (1998) observed that EC and Mg<sup>++</sup> were significantly higher (p < 0.05) in the first soil zone 0-15 cm, whereas they were higher in soil zone 0-30 cm particularly in calcareous soil (Kiziloglu, et. al., 2008). According to Heidarpour et al. (2007) EC increased in the upper soil layer resulting in inhibition of plant growth after sub-irrigation with wastewater. However, Mollahoseini et al. (2013) reported that EC did not show any significant differences (p>0.05) in soil laver (0-20 cm) but it increased (p < 0.05) in soil layer (20-40 cm) between (1 ds/m–1.6 ds/m respectively) after 30 years of irrigation with wastewater. In addition, an EC increase was observed in deeper soil layers with a tendency to be higher after a longer irrigation period. This is due to the soluble salts accumulation in deeper layers after draining (Abu-Awwad, 1996). In a clay soil, the EC increased accordingly in the soil layer above drip lines but the range was significantly lower than the threshold of soil salinity after sub-irrigation with wastewater (Jiajie Hea et al., 2013).

## 4. Conclusions

After irrigation with U untreated and T treated wastewater in SL and LS soils, results lead to the following conclusions: pH and sodium decreased whereas phosphorus and electrical conductivity increased. The other examined parameters were as follows: in SL soil, CaCO3% decreased while potassium remained unchanged. In LS soil, CaCO3% increased but potassium decreased. Statistical analysis of the above parameters showed that these were not statistically significant (p> 0.05). However, statistically significant differences (p < 0.05) were observed in Na<sup>+</sup>  $\mu$ g/g, and electrical conductivity (EC), but only for the LS soil.

Between the two zones (I) and (II) at depths of 10 and 20 cm sub-irrigation, statistically significant differences were found as follows (p < 0.05): In SL soil, there was an increase in phosphorus (irrigation with U at 10 cm) and in sodium (irrigation with T at 20 cm) in the upper zone (I). In LS soil in zone (I), there was an increase in potassium (irrigation with U at 10 cm) and in electrical conductivity (irrigation with T at 20 cm) while in zone (II) there was an increase in CaCO3% (irrigation with T at 10 cm) and in pH (irrigation with U at 20 cm). Phosphorus potassium and electrical conductivity increased in the upper zone (I) in two soils SL and LS, while CaCO<sub>3</sub>% and pH increased in the lower one zone (II) only in LS soil.

Between SL and LS soils, it can be concluded that wastewater use for irrigation may lead to an increase in sodium and electrical conductivity in LS soil (loamy sand). Generally, it seems that Loamy sand soils are more affected by irrigation with untreated and treated wastewater use. Therefore, irrigation with wastewater (especially untreated) could lead to an improvement of the soil properties, but caution is needed because wastewater use can cause an increase in soil salinity and electrical conductivity after long-term irrigation.

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