

# Effect of Treated Wastewater Irrigation on Heavy Metals Distribution in a Tunisian Soil

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**Abstract**—Treated wastewater (TWW) may contain toxic chemical constituents that pose negative environmental and health impacts. In this study, soil samples under treated wastewater irrigation were studied. For this purpose, six plots were made in an irrigated area in north of Tunisia and treated with two water qualities: fresh water (FW) and treated wastewater (TWW). Five soil depths were used: 0-30, 30-60, 60-90, 90-120 and 120-150 cm. The TWW irrigation increased significantly ( $P \leq 0.05$ ) the soils' EC, Na, K, Ca, Mg, Cl, SAR, Cu, Cd and Ni and had no significant ( $P \leq 0.05$ ) effect on the soils' pH, Zn, Co and Pb contents. EC, Na, Cl, SAR, Zn and Co increased significantly with soil depth. The results for K, Ca, Mg, Cd, Pb and Ni exhibited similar repartition in different layers of soil. It was also shown that the amount of different elements in soil irrigated with fresh water (FW) were less compared with the control soil.

**Keywords**- TWW; FW; soil; salinity; Heavy metals; Tunisia

## I. INTRODUCTION

In many arid and semi-arid regions, natural fresh water (FW) resources are limited, whereas the demand is constantly increasing due to industrial and population growth. The greater challenge to meet water demands and manage its limited natural resources has led to the use of alternative irrigation sources [1]. One of these alternatives is to use non-conventional water resources, such as Treated Waste Water (TWW). The reuse of TWW for purposes such as agricultural irrigation has become, for many countries, an important element in water resources planning [2]. TWW can be used to help in reducing natural water consumption, in restoring and preserving degraded land and in aiding the growth of vegetation [3-7].

The reuse of TWW for irrigation is considered an environmentally sound wastewater disposal practice compared to its direct disposal to the surface or ground water bodies. In

addition, TWW provides convenient disposal of waste products [8] and constitutes a significant source of plant nutrients and organic matter needed for maintaining the fertility and productivity levels of the soil [9, 10]. However, besides these beneficial effects, TWW is often associated with environmental and health risks. The main health risks are associated with the contamination of crops or ground waters by TWW due to its chemical composition being somewhat different from most natural waters used in irrigation [11]. This water may contain high levels of salts, toxic ions, heavy metals, and organic residues. Accumulation of these pollutants in soil can be harmful in fields irrigated for long periods of time and poses a threat to agricultural production and the environment [12].

Several studies have reported some changes in the physicochemical characteristics of soil due to TWW application [13, 14] such as the increase of soil salinity, electrical conductivity (EC), organic matter, exchangeable Na, K, Ca, Mg, plant available phosphorus and microelements [14-17] and the decrease of soil pH [16]. In general, heavy metals combine with other anions or anionic compounds to form stable substances and tend to accumulate in the topsoil (0-35 cm). Furthermore, their uptake by plants increases with decreasing pH, due to the dissolution of metal-carbonate complexes that releases free metal ions into the solution [18]. However, when the capacity of soils to retain toxic metals is reduced due to continuous loading of pollutants or changes in pH, metal ions that have a relatively high mobility can migrate in depth and contaminate groundwater by percolation into the ground [19].

The purpose of the present work is to study and compare the effects of FW and TWW irrigation on a Tunisian clay loam soil properties and its macro- and micro-nutrients, and heavy metal content, in order to establish the basis for safe TWW agricultural reuse.

## II. MATERIALS AND METHODS

## A. Experimental design

The experiment was located in Cebela Borj-Touil field located 20 km north of Tunisian the Low Valley of Medjerda. Climate of the region is semi-arid with an annual rainfall close to 470 mm and an average yearly evapotranspiration of 1400 mm. The soil is clay-loam. The experiment included two sources of irrigation water: (a) TWW and FW (b). The experiment was carried out in strip plot design with three replications and a total of  $2 \times 3 = 6$  plots of  $3 \text{ m} \times 3 = 9 \text{ m}^2$  size. The experimental plots were irrigated by wastewater from Chotrana treatment plant located in north of Tunis city.

The TWW and FW were applied during the summer period. In each plot, soil samples were collected from five successive depths 0-30, 30-60, 60-90, 90-120 and 120-150 cm. Control soil samples were collected in the beginning of the experiment. Water samples were collected periodically during the experimental period and the chemical characteristics are summarized in Table I.

TABLE I. CHEMICAL PROPERTIES OF FW AND TWW

Parameter	Irrigated water		INNORPI (NT-1989)*
	FW		
pH	8.5b	6.5-8.5	6.5-8.5
EC (dSm <sup>-1</sup> )	1.42a	7	7
SO <sub>4</sub> <sup>-</sup> (mg l <sup>-1</sup> )	9.47a	-	-
Na <sup>+</sup> (mg l <sup>-1</sup> )	6.45a		
Ca <sup>++</sup> (mg l <sup>-1</sup> )	6.28a	-	-
Mg <sup>++</sup> (mg l <sup>-1</sup> )	4.91a	-	-
Cl <sup>-</sup> (mg l <sup>-1</sup> )	9.39a	2000	2000
K <sup>+</sup> (mg l <sup>-1</sup> )	0.14a		
Cd (mg l <sup>-1</sup> )	0.045a	0.05	0.05
Co (mg l <sup>-1</sup> )	0.033a	0.1	0.1
Cu (mg l <sup>-1</sup> )	0.019a	0.2	0.2
Ni (mg l <sup>-1</sup> )	0.06a	0.2	0.2
Pb (mg l <sup>-1</sup> )	0.16a	5	5
Zn (mg l <sup>-1</sup> )	0.010a	2	2

\*Tunisian Standard

## B. Soil sampling and analysis

Collected soil samples were transported to the laboratory, oven, dried at 105° C and sieved for chemical analysis. The pH and the electric conductivity (EC) were respectively measured with pH-meter and conductivity-meter in a soil-water suspension.

The sodium absorption ratio (SAR) [20] was calculated by the following formulae:

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

Concentrations of Ca and Mg were measured using the EDTA titration method. Na and K contents were measured using flame photometer [20]. Soil samples were digested for heavy metal (Cu, Zn Cr, Cd, Ni and Pb). The procedure based

on acid digestion induced by microwave energy was optimized in order to measure the total heavy metals contents in soils. Each soil sample (1 g) was placed in a Teflon vessel (100 mL) with HF (10 mL) and HClO<sub>4</sub> (5 mL) and then digested in a microwave. After that, 70 ml of perchloric acid was added to the mixture. After digestion, the samples were filtered and transferred into 100 ml volumetric flasks and brought to volume of 100 ml by added of distilled water. The filtrate was analyzed by atomic absorption for the determination of the following: Mn, Cu, Fe, Pb, Co and Ni (ISO 14869-1, 2001).

## C. Statistical analysis

The statistical analyses were performed for each parameter in each soil layer and for different treatment. The data were subjected to one-way analysis of variance (ANOVA) using STATISTICA software, Version 5 (Statsoft France, 1997). Mean comparison was carried out using LSD test at the significant level of 0.05.

## III. RESULTS

## A. Soil pH

As showed in Table II, pH of soil has not been significant altered in the treated soils. The pH is low in the depth of soil of 90-120 cm. A low value of pH was noted in all treated soils.

## B. Soil Electrical Conductivity (EC)

According to Table II, the soil EC was significantly increased in two depths (30–60 and 60–90 cm). This increase was, respectively, from 2.72 to 4.49 and 7.9 to 8.21 dSm<sup>-1</sup>. The data obtained after FW application indicated that EC was decreased, respectively, from 6.13 to 4.38 and from 7.91 to 5.78 dSm<sup>-1</sup> in 90-120 and 120-150 cm soil depth.

TABLE II. EFFECTS OF TWW ON PH AND EC OF SOIL

Parameter	Soil layers (cm)					
	Treatment	0-30	30-60	60-90	90-120	120-150
pH	Control	8 bcd	8 bcd	8,21 cd	7,71 ab	8,17 c
	FW	8 bcd	8 bcd	8,2 cd	7,7 ab	8,2 cd
	TWW	7,9bcd	8bcd	8,17 cd	7,58 ab	8,2 cd
EC	Control	2,73 a	2,72 a	3,53 ab	6,13 c	7,91 d
	FW	2,73 a	2,72 a	3,51 ab	4,38 b	5,78 c
	TWW	3,25 a	4,49 b	5,94 c	6,13 c	7,97 d

## C. Major elements contents

The results for Na, K, Ca, Mg and Cl are summarized in Table III. The TWW treatment led to an increase in Na content in the soil particularly at the depth of the 0-30 cm. In comparison to the control treatment, the Na content was increased from 19.5 to 32.75 meq.L<sup>-1</sup>. For K content, the TWW treatment applied increased significantly the K content in a soil depth of 0-30 cm compared to the control and FW treatment of soil. The K amount in the control soil was 0,34 meq.L<sup>-1</sup> and was increased significantly to 4.47 meq.L<sup>-1</sup> with the irrigation with TWW. The Ca content in soil irrigated with TWW was significantly higher in the soil depth of 0-30 cm compared to the control treatment. The application of TWW led to an

increase of Mg content especially in the soil depths of 0-30 and 90-120 cm. As showed in the Table III, the Cl content was greater in soil depths of 0-30 and 90-120cm with the TWW application.

#### D. Soil sodicity (SAR)

As shown in Table III, the SAR values were not significantly affected by the different waters qualities. However, in all treatments, the SAR level increased significantly with the increasing of soil depth. .

TABLE III. OIL CHEMICAL CHARACTERISTICS

Parameter	Treatment	Soil layers (cm)				
		0-30	30-60	60-90	90-120	120-150
Na	Control	19,5 <sup>a</sup>	27 <sup>ab</sup>	45,79 <sup>de</sup>	54,05 <sup>efg</sup>	61,15 <sup>g</sup>
	FW	17,17 <sup>a</sup>	19,4 <sup>a</sup>	41,3 <sup>cd</sup>	53,3 <sup>defg</sup>	51 <sup>defg</sup>
	TWW	32,7 <sup>bc</sup>	29 <sup>ab</sup>	48,2 <sup>def</sup>	54,56 <sup>fg</sup>	61,44 <sup>g</sup>
K	Control	0,34 <sup>a</sup>	0,09 <sup>a</sup>	0,17 <sup>a</sup>	0,11 <sup>a</sup>	0,17 <sup>a</sup>
	FW	0,11 <sup>a</sup>	0,07 <sup>a</sup>	0,1 <sup>a</sup>	0,11 <sup>a</sup>	0,13 <sup>a</sup>
	TWW	4,47 <sup>b</sup>	0,79 <sup>a</sup>	1,75 <sup>a</sup>	1,28 <sup>a</sup>	1,16 <sup>a</sup>
Ca	Control	6,7a <sup>bc</sup>	6,1a <sup>b</sup>	8,7 <sup>abcd</sup>	14 <sup>c</sup>	11,4 <sup>cde</sup>
	FW	5,44 <sup>a</sup>	5,03 <sup>a</sup>	8,1 <sup>abcd</sup>	8,23 <sup>abcd</sup>	8,56 <sup>abcd</sup>
	TWW	11 <sup>bcd</sup>	6,7 <sup>abc</sup>	10 <sup>abcde</sup>	14,14 <sup>c</sup>	12,93 <sup>de</sup>
Mg	Control	3,22 <sup>a</sup>	5 <sup>abc</sup>	6,64 <sup>cde</sup>	8,38 <sup>cde</sup>	8,8 <sup>de</sup>
	FW	5 <sup>abc</sup>	3,7 <sup>ab</sup>	6,5 <sup>abcd</sup>	8,26 <sup>cde</sup>	7,6 <sup>cde</sup>
	TWW	7,6 <sup>cde</sup>	5,2 <sup>abc</sup>	6,73 <sup>bcd</sup>	10,29 <sup>e</sup>	9,29 <sup>de</sup>
Cl	Control	15,68 <sup>a</sup>	22 <sup>ab</sup>	30,22 <sup>bc</sup>	54,75 <sup>ef</sup>	54,14 <sup>ef</sup>
	FW	14,77 <sup>a</sup>	14,9 <sup>a</sup>	34,6 <sup>abc</sup>	45,47 <sup>def</sup>	45,24 <sup>def</sup>
	TWW	30,3 <sup>bc</sup>	24 <sup>ab</sup>	41,7 <sup>cde</sup>	74,77 <sup>f</sup>	58,01 <sup>f</sup>
SAR	Control	8,3 <sup>a</sup>	10,8 <sup>a</sup>	17,35 <sup>b</sup>	17,41 <sup>b</sup>	18,85 <sup>b</sup>
	FW	7,87 <sup>a</sup>	9,33 <sup>a</sup>	15,58 <sup>b</sup>	16,40 <sup>b</sup>	18,01 <sup>b</sup>
	TWW	10,77 <sup>a</sup>	11,9 <sup>a</sup>	16,45 <sup>b</sup>	18,57 <sup>b</sup>	19,0 <sup>b</sup>

#### E. Heavy metal concentrations in soils

##### 1) Zinc (Zn)

According to Figure 1a, the Zn content in the soil samples irrigated with TWW was higher in the soil depth of 90-120 cm and didn't show a significant change in others soil depths compared to the control soil.

##### 2) Cobalt (Co)

As shown in Figure 1b, the TWW treatment had a significant effect on Co content with the soil depth in comparison to the control treatment. The maximum Co content was 30.63 mg.kg<sup>-1</sup> and was found in the soil depth of 90-120 cm.

##### 3) Copper (Cu)

The TWW application increased significantly the Cu content with the soil depth in comparison with the control treatment. The highest increase was detected in the soil depth of 30-60 and 90-120cm. This increase was from 8.56 and 8.1 mg.Kg<sup>-1</sup> to 16 and 16.66 mg.kg<sup>-1</sup> respectively (Figure 1c).

##### 4) Cadmium (Cd)

The results in Figure 1d showed a high accumulation of Cd contents in the case of TWW treatment in all soil depths compared to Cd content in the control soil and soil treated with the FW.

##### 5) Plomb (Pb)

Results shown in Figure 1e indicate that TWW application increased significantly the Pb concentration (126 mg.kg<sup>-1</sup>) in the soil depth of 0-30 of TWW irrigated soil as compared to control soil. While Pb concentration decreased due to irrigation with FW in all soil layers.

##### 6) Nickel (Ni)

As presented in Figure 1f, TWW application to the soil led to significant increase of Ni content in all soil depths compared to the control soil and the soil treated with FW. Indeed, the highest levels of Ni, with an average of 105.33 mg kg<sup>-1</sup>, were found the first soil depth (0-30 cm).

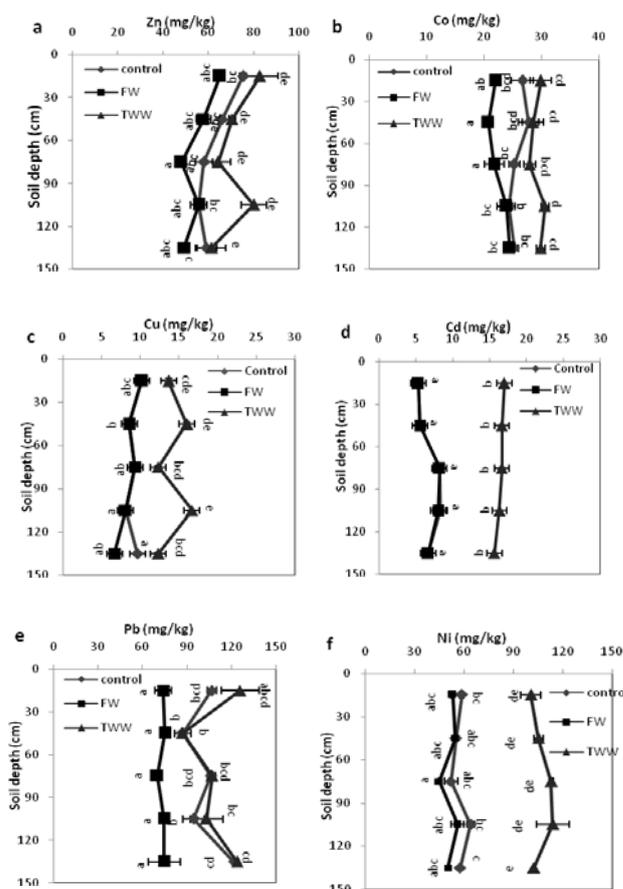


Fig. 1. Concentration of heavy metals (mg/kg) in the soil layers at the beginning of experiment and after irrigation with TWW and FW.

#### IV. DISCUSSION

Soil pH is well known for his important role in the mobility of metals as in their bioavailability for plants [21, 22]. Indeed, metal availability is relatively low when pH is around 6.5 to 7, as for lower pH would favor availability, mobility and redistribution of the metals. In the present study, result showed that the pH is around 7.5 and 8.2 and this statement limited the mobility of metal. Although irrigation with TWW led to a slight decrease of soil pH with depth, the soil was alkaline. Some investigations showed that irrigation with wastewater

decreased the soil's pH. This decrease was due to the decomposition of organic matter and production of organic acids. Others authors [23, 24 25] found that irrigation with TWW raised the soil pH, respectively, by 1.3 and 0.2 U of pH. Other researchers [10, 11] explained this increase by the long term impact of irrigation with sewage and wastewater effluents on soil properties.

Results indicated that the electrical conductivity of soil irrigated with TWW increased significantly compared. Similar results were reported in [10, 12-13]. This increase has been attributed to the higher concentration of cations such as Na and K in wastewater [10]. This increase concerned soil depths of 30–60 and 60-90 cm. However, FW irrigation led to significant decrease of EC. In a similar way, Al-Nakshabandi et al. [14] explained this decrease by the leaching and displacement of salt after irrigation.

The results for Na showed high concentrations in the upper soil layer of TWW irrigated soil. This is in line [6]. However, a significant decrease of Na level was recorded in the first layer of FW irrigated soil. This decrease is probably due to the leaching of soluble Na into deeper soil layers [15].

The K concentration in TWW irrigated soil increased significantly compared to the control and FW irrigated soils. These results were in accordance with [13] but not in agreement with [16] that reported a decrease of K soil levels with TWW irrigation and explained this by root uptake and the leaching of K<sup>+</sup> into deeper soil. The concentrations of Ca in the first soil layer (0-30) of TWW irrigated soil samples were significantly higher than those in FW irrigated soil. Similarly, Heidarpour et al. [6] reported that irrigation with wastewater generated a greater Ca concentration than groundwater. They explained this statement by the difference in plant yield using two different irrigation waters and consequently the effect of plant uptake on the soil solution. FW led to a significant decrease on the average of Ca in most of soil layers. This reduction in soil Ca could be due to leaching, plant uptake and reaction of Ca with carbonate and sulfate, which were present in the water [6]. As for Mg, TWW increased significantly the Mg concentration in the first layer of soils compared to control soil in agreement with [6]. However, a significant decrease (of Mg level occurred in the case of the FW irrigation. This reduction has been reported as a possible in [1].

For Cl content, TWW caused significant increase of this element in soil. Similar results have been reported in [13, 17]. In general, the Cl increased with increasing soil layers for both irrigated and control soils. Similarly, it has been reported that chloride is usually not adsorbed or held back by soils but it moves easily with soil water [16].

Both TWW and FW irrigation had no significant effect on SAR. The results showed an increase of SAR with depth. This significant increase could be explained by the leaching of Na<sup>+</sup> into deeper soil. This increase below soil layers disagrees with the findings of [18] which noted that SAR diminished with increasing depth. The high level of SAR (>13) indicated that TWW irrigation increased the soil sodicity.

Result showed that Cu concentration was significantly higher in the TWW irrigated soil than in the control soil. Our

results corroborate with these obtained in [16]. On the other hand, it has also been reported in [2] that the application of wastewater had no significant effect on the amount of Cu compared with the beginning stage and with the groundwater treatment. In fact, Saber in [26] showed that a seven-year application of wastewater had no significant effect on the concentration of Cu in the soil. Also, Adriano [27] stated that Cu is stabilized in soil by clay minerals, organic matters and Fe, Al and Mn oxides. The vertical distribution changed over time with alternation of increase and decrease trend. This tendency agrees with [28] where it is reported that Cu has certain mobility from upper horizons to lower horizons.

The Co level increased significantly in TWW irrigated soil. These results were in accordance with [2] where it is reported that TWW irrigation had a significant increase on the amount of Co compared with the beginning of the growing season and with the groundwater treatment. In [29] it was reported that application of wastewater for irrigation for a period of 47 years caused a significant increase of the total and available Co in soil. In general, the accumulation of Co increases with increased soil depth and these results were in accordance with those in [2].

As for Zn, application of TWW had no significant effect on the accumulation of Zn. These results are in agreement with findings obtained previously [2]. However, a significant increase of Zn concentration was shown in 90-120 cm depth. These results contradict the findings in [30] where a significant accumulation of Zn in the upper (25-30 cm) soil with wastewater irrigation was reported. This increase might be attributed to the leaching and displacement of Zn by irrigation into upper soil layers. We can, also, deduce that Zn concentration in FW irrigated soil decreased compared with the control soil.

Application of TWW increased significantly the Cd content. Similar results were observed in [31] where it is reported that the concentration of Cd in soil irrigated under wastewater was increased. On the other hand, in [10, 20, 32] it was shown that irrigation with wastewater does not effect the concentration of cadmium. Overall, the Cd levels in various soil depths were similar and no tendency was noted. These results were in concordance with those obtained in [33].

Result for Pb showed that, with the exception of the surface layer, TWW irrigation had no significant effect on the soil. Similar results were reported in [2, 10]. Moreover, no significant effect was noted regarding depth. However, many authors have shown a significant decrease of Pb through the soil layers [33, 34]. The Pb content was reduced in FW irrigated soil. These results might be explained by the lixiviation of the element in soil due to precipitation events after irrigation season [35, 36].

In the case of Nickel, soil irrigated with TWW exhibited significant decrease of this metal in comparison with the control soil. These results were in disagreement with [19]. Ni level was higher in all depths of TWW irrigated soils compared to both other cases (control and FW treatment). This is in line with finding reported in [37]. According to our study, no differences were found for soil depth expected the slight

increase of Ni in the TWW soil in the depth of 90-120 cm. In similar way, McLaren et al. [28] found that about 57% of Ni applications were lixiviated from upper soil horizons to lower soil horizons.

Based on our study, the concentration of heavy metals (Zn, Co, Cu, Cd, Pb and Ni) was higher in most of depths of TWW irrigated soil compared to those of FW irrigated soil. It has been reported that high concentration of heavy metals in wastewater leads to an increase of their content in soil [31, 37-39].

Heavy metals are priority toxic pollutants that severely limit the beneficial use of water [40]. Soil may adsorb and retain important amount of heavy metals from wastewater. In this study, the comparison of Zn, Cu, Pb and Ni with a standard level of heavy metals in soil showed that Cd and Ni of control soil and soils irrigated with the both TWW and FW were higher than their maximum permissible limit (3 and 50 ppm) in soil by USEPA ([41]). It can be predicted that Cd and Ni have been, probably added to the water and soil from other sources. The results for Zn, Cu, Pb were far less than the USEPA maximum permissible limits (50, 200 and 300 ppm). The contamination of soil by some heavy metals as Cd and Ni presents a worrying situation that should be monitored to prevent further environmental and health risks.

#### V. CONCLUSION

This study contributes to the evaluation of TWW effects on clay-loam soil properties, in order to test the possibility of TWW safe reuse. Indeed, after one cycle of irrigation, we found some variations in the soil properties as a result of TWW application. We have detected an increase in EC, Na, K, Ca, Mg, Cl, SAR, Cu, Cd and Ni content in soil irrigated with TWW in comparison with soil irrigated with FW. However, no remarkable changes in soil pH, Zn, Co and Pb was shown. We saw a significant increase in EC, Na, Cl, SAR, Zn, Co and Ni level with depth. But, no significant change in pH, K, Cu, Cd, Pb and Ni was shown, for different depths. The results of this study show that TWW reuse in irrigation must be conditioned by some management measures such as soil texture, plant selection and the choice of irrigation methods.

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