

Study of heavy metals absorption in rosebushes and soil irrigated with reclaimed wastewater

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Abstract— Proper wastewater treatment and their application in agriculture can bring great benefits to plants, as well as for the environment. Such practice may properly serve as an alternative to the high demand for water in the agricultural sector. But the reclaimed wastewater can contain potentially toxic metals and if used improperly may bring in crop losses and damage farmers workers and the environment. This study evaluated the impacts on soil and on rosebush of the use of a reclaimed wastewater with a high content of metal. That reclaimed wastewater was coming for the university hospitalar complex, and before the application on soil was treated by a simplified system. The rosebush and soil used had the same management that is used in rose farms in region. this study demonstrated that the application of this effluent in agriculture did not bring damage to the soil and the plant

Index Terms— Heavy metals; fertigation; environmental impacts.

I. INTRODUCTION

Water scarcity is a reality that affects every continent on the planet e can be seen as a major problem to be faced by society [1]. Worldwide agriculture consumes about 85% water used by man activities [2]. Close to that, the last decade has seen a decline in per capita availability of water and it is expected that this availability will continue to decline due to population and economic growth [3].

In the big cities, in most cases, there is no way to increase the production of treated water due its scarcity or it is not economically viable to seek new water sources. Thus, the increasing demand for water by both industrial facilities and urban areas has led to a decrease in the allocation of water for agriculture. So, some providences must be taken, and among the possible solutions is the improved irrigation technologies and reuse of non-conventional water resources [4]. The use of reclaimed wastewater as water source for irrigation is an alternative to counteract water scarcity. Allied to this, the treated effluent also has great potential benefices to plants, since it is rich in nutrients necessary for the development of agricultural crops [5].

However, although the reuse of wastewater in agriculture can increase agricultural production and expand the cultivated lands some contaminants in the wastewater may affect the soil causing changes in its composition which may generate some environmental impacts [6]. Some studies have reported the risk of accumulation of heavy metals in topsoil [7] which can cause losses in crop yield or the accumulation of metals in plants causing problems in human health [8]. It is of great importance to monitor the soil and the plant to quantify the interference of heavy metals presents on reclaimed wastewater on irrigation.

In agriculture, cadmium contamination in farmland has seen significant growth since the last century, causing many health problems [9]. In soil, the metals are retained on the top when pH is above 6.5 and when there is a large amount of organic matter. When the pH drops of that valor, the metal may became soluble and can be absorbed by the plant or be leached [10].

Although the application of reclaimed wastewater may be source of heavy metals into the soil, the conventional management also generates impacts as the heavy metals. Organic and inorganic fertilizers are potential sources of heavy metals in agricultural soils [11]. The increase in content of heavy metals in the soil and the plant has already been verified in studies in which both chemical fertilizer was used in excess or as recommended to agriculture [12], [13]. Caution should be taken at this point, since in fact, large amounts of fertilizer and pesticides are used in practice by farmers aiming to achieve the maximum productivity, which may culminate in the accumulation of heavy metals in the soil, such as cadmium which is present for example in lime and superphosphate [14].

So, the aim of this study was to evaluate the accumulation of heavy metals in soil and plants irrigated with treated effluent hospital complex of a simplified system of wastewater treatment.

II. MATERIALS AND METHODS

The raw wastewater was coming from the university hospital complex. This wastewater was directed to four anaerobic filters built in stainless steel cylindrical containers with a total volume of 500 L, and operated with upflow hydraulic retention time of 9 hours. The support material used was composed of coconut shells from the *Cocos nucifera* species which had each unit divided into four parts prior to its placement within the cylinder. The effluent generated by these reactors followed two distinct paths in the experimental

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Table 1 - Summary of all water types used to irrigation and fertilization

Test	type of management	Number of beds
Control	Out of greenhouse - reclaimed wastewater without fertilization and agrototoxic	4
T1	Clean water without fertilization	4
T2	Reclaimed wastewater from sand filter + fertilization	4
T3	Reclaimed wastewater from anaerobic reactor + fertilization	4
T4	Reclaimed wastewater from anaerobic reactor	4
T5	Reclaimed wastewater from sand	4
T6	Clean water + fertilization	4

area: a) half was directed to tanks for the rosebushes' irrigation (anaerobic effluent irrigation); b) half was applied on the surfaces of four intermittent sand filters, at the rate from 300 L m⁻² day⁻¹ to 800 L m⁻² d⁻¹, and in loads of 50 L m⁻² evenly distributed throughout the day (nitrified effluent), for the subsequent irrigation of a different group of rosebushes.

Fiber glass cylindrical boxes with internal diameter of 1.00 m were used in the construction of the sand filters. The bed was composed by three stratified layers from the base of the reactor. The first was 0.20m deep and consisted of gravel with effective size (D10) of 16.12 mm and uniformity coefficient (UC) of 1.88. The second layer consisted of gravel with D10 equal to 7.51 mm and UC of 1.66 C, and was 0.05 m deep. This material aimed to support the sand; thus, preventing the drainage of its particles.

The sand bed was 0.75 m deep, and the sand used had effective size of 0.17 mm and uniformity coefficient of 3.14. After passing through the sand filter, the effluent showed a complete nitrification of the nitrogen compounds, and it was transferred to tanks for the rosebushes' irrigation.

The chosen culture was the Rosa hybrida ambiance variety. The culture was transplanted into 24 beds of 5.4 m² and 0.40 m deep. The rosebushes was spaced 0.15 m, with a total of 21 plants in each row (total of 3 row). For protection of rosebush was built a greenhouse arch type, with transparent cover of low density polyethylene. The greenhouse was 576 m²

The soil utilized by this study was classified as sand clay, with density of 1.3 g cm⁻³, field capacity with 26.13% and wilting point of 17.18%. Chemical soil analysis was also performed, leading to the need of correction of acid with dolomitic limestone before transplanting rosebushes. In the setting process was upturned limestone to a depth of 0.20 m. the experimental design was randomized blocks, as shown in Table 1

Fertilization was performed at 30, 75 and 110 days after planting for T2, T3 and T6 treatments, using NPK (10:10:10) and ammonium sulfate. This action fought to evaluate the need for topdressing for growing rose with the use of irrigation with effluent under study. Both topdressing as planting fertilization were performed according to recommendations [15] Clean water for irrigation for T3 and T4 treatments came from the municipal supply.

Irrigation was set according to the water requirement of the culture, which was determined based on the tensiometers installed in the central portion of each bed in two different depths: 0.10 and 0.30 m. irrigation was initiated when the pressure of tensiometers was -10 kPa, using 1.3 mm polyethylene drip tubes, providing a flow rate of 1 L h⁻¹.

Rosebush variety used in this study was susceptible to infestations by pests and fungi. Thus, we noted the presence on roses of mildew fungus and aphids. In order to keep growing, the research group visited several farms producing cut roses in the region which is the largest producer and exporter rose flowers from Brazil. So, the producers were consulted, and we used their management to pest control.

Therefore, as the farms that produces roses commercially, in this study some agricultural pesticides were used. Against invasive plants it was used glyphosate. For control of fungi, it was used three fungicide whose active ingredient are: Chlorothalonil, Zinc ammoniate ethylenebis (dithiocarbamate) – poly[ethylenebis (thiuramdisulphide) and Boscalid + Kresoxim-Metil. To the control of aphids was used active ingredient: alphacypermethrin e Imidacloprid. The rosebush was sprayed with fungicides twice weekly alternating the active principle and to control of aphids whenever there was the appearance of these.

At the end of 36 months the soil sampling was conducted in fifteen points per treatment of topsoil and in depth from 0.00 to 0.20 m. It was also collected soil from the outside of the greenhouse, which received no irrigation and spraying pesticides. The roses leaves, stems and petals were also collected.

From the samples it was analyzed the content of heavy metals using microwave digestion by EPA method 3051 and reading on atomic absorption spectrophotometer.

III. RESULTS AND DISCUSSION

The analysis of heavy metals content in the reclaimed wastewater used in this study are shown in Table 2.

The values found for the parameter studied were compared with Brazilian legislation with provides the criteria for effluent discharge into receiving water bodies and only the elements cadmium and copper were within the range specified by that legislation. The highest concentration was found for zinc with the average nine times higher than the amount allowed in the effluent from anaerobic filter and 38 times higher in the effluent from sand filter. Lead, chromium and zinc presented values well above the allowed and can be problematic to the soil-plant system. Still under Brazilian law the values of lead and zinc are above the maximum allowed in liquid effluents from treatment stations for use in agriculture.

Table 3 presents the values found for heavy metals in the soil from the beds in comparison to that in the initial conditions of the study. For cadmium the highest values were found in treatments T1 and T2, whose concentration was 1.8 mg kg⁻¹. This value was statistically different from the

Table 2 - Concentration of heavy metal in the irrigation water

	Metals (mg L ⁻¹)				
	Cadmium	Lead	Copper	Chromium	Zinc
Clean water	<0,001	<0,001	<0,001	<0,001	<0,001
Effluent from anaerobic filter	0,005±0,007	1,346±0,701	0,581±0,128	13,926±2,843	45,500±22,941
Effluent from sand filter	0,004±0,005	1,482±0,765	0,606±0,089	13,877±1,719	193,500±105,478

Table 3 - values found for heavy metals in the topsoil (0.00 to 0.05 m)

	Cd	Pb	Cu	Cr	Ni	Zn
	-----mg Kg-1-----					
Co	<0,1	0,3	2,7	<0,1	<0,1	0,2
T1	1,8 ±0,1a	12,0±1,0a	43,7±7,4a	81,4±9,2a	7,2±1,8a	21,6±3,1a
T2	1,8± 0,1a	12,2±0,9a	45,2±9,7a	82,7±10,6a	8,0±2,5a	22,2±7,9a
T3	1,3±0,4b	9,2±1,8a	36,1±0,3a	64,6±10,6a	5,0±0,3a	19,3±3,7a
T4	1,2±0,2b	8,9±1,5a	33,6±4,2a	58,6±7,3a	4,6±0,7a	14,6±4,0a
T5	1,3±0,2b	9,6±1,4a	35,2±2,5a	66,7±9,6a	7,0±3,0a	13,6±2,1a
T6	1,2±0,2b	9,6±0,9a	36,8±2,0a	67,3±5,9a	5,8±0,7a	14,2±4,4a

Co - Initial soil condition

*Statistical analysis between values with different superscript letters are statistically significant ($p < 0.05$)

values obtained in other treatments. The average concentration of cadmium in soils varies between 0.6 and 1.1 mg.kg⁻¹ [16] and the value found in all treatments over the range of common values.

As the contents of other metals (lead, copper, chromium, nickel and zinc) in all treatments were statistically significant differences between them. Levels of lead found ranged from 8.9 to 12.2 mg kg⁻¹ values that are within the reference limits for quality. This value is well below the worldwide range of soils that Pb is 10 to 150 mg kg⁻¹ [17]. Copper presented concentrations between 33.6 and 45.2 remaining within the limits of prevention. Copper concentrations are found in the global range which covers the soil is 1 to 140 mg kg⁻¹ [16]. Chromium in the treatments ranged between 58.6 and 82.7 mg kg-1 and were within the limits of prevention in the treatments T3, T4, T5 e T6. T1 e T2 were classified as above the limit of prevention, but they did not have reach the intervention limit to agricultural soils. The worldwide average concentration of chromium in the soil is 54 mg kg⁻¹ [16], and can vary between 20 a 200 mg kg⁻¹ [17].

Nickel and zinc, in all treatments showed concentrations values within the limit of the reference quality. These values are also within the limits of the world average. The concentration of nickel in soils varies quite markedly, but the world average concentration is 40 mg kg⁻¹. Zinc average concentration is in the range of 43 mg kg⁻¹ [16] and all values found in the soil in this study between theses averages.

For all metals analyzed there was a marked increase comparing the initial conditions of the soil before planting. However, this increase occurred equally for all treatments, regardless of the water used for irrigation or fertilization management adopted. According to [18] the spraying of pesticides and agrotoxics in agricultural crops increases the surface concentration of heavy metals in the soil, since these elements form part of the pesticide composition. The rosebush variety used in the study was very susceptible to attack by fungi and aphids, which made the necessary constant spraying throughout the experiment. Such sprays were responsible for increasing the concentration of heavy

metals in soil, once the experiment occurred in a closed environment without suffering the leaching action of rainfall.

Reference [19] found higher values of heavy metals in soils which were cultivated citrus irrigated over 11 years with reclaimed wastewater. In that study, copper, nickel, cadmium, chromium and lead were much higher when using the effluent for irrigation achieving increasing concentrations of these metals 20, 143, 19, 58 and 92% respectively, values which however did not exceed the limits common.

Table 4 presents the average values of metals found in soil collect depth from 0.00 to 0.20 m

Although the concentration of heavy metals in soil has increased in the topsoil in the deep layer (0.00 a 0.20 m) the concentration was lower. In this case, the average concentrations of metals found in the soil were below the reference values of quality and prevention arranged in Brazilian legislation [20].

These data indicate, in a first analysis, that nevertheless the reclaimed wastewater had a high content of heavy metals, above the allowed limits on Brazilian legislation, its use on irrigation did not cause soil contamination on the deep layers of the soil where the rosebush was cultivated.

Also in Table 4 it can be seen that for all treatments the presence of heavy metals was similar. For the control treatment the soil did not received agrotoxic and pesticide application and presents values below the other. It can be inferred that the application of pesticides causes and increase in concentration of heavy metals in the soil, far more significant the application of effluent.

Table 5, Table 6 and Table 7 show the average values of heavy metals found in rosebush petals, leaves and stem.

In the three analyzed parts of the rosebush, the values found for Cu, Cd, Pb, Ni e Cr were below detection limits for all treatments studied. For Zn and Ni the values found in the petals, leaves and stems were also low, bur suffered variances between treatments.

In petals, the treatment in which effluent from sand filter was used presented the highest metal content in relation to others. Irrigated roses with effluent from anaerobic reactor

Table 4 - Average values of metals in soil in the 0.00 to 0.20 m

Treatment	Cu	Cd	Zn	Pb	Ni	Cr
mg Kg ⁻¹						
Control	0.06a	0.02	0.41 a	0.10a	0.19a	0.04a
T1	0.32 ± 0.02b	BDL	0.55 ± 0.11b	0.25 ± 0.02b	0.31 ± 0.02b	0.40 ± 0.01b
T2	0.34 ± 0.04b	BDL	0.53 ± 0.19b	0.26 ± 0.03b	0.28 ± 0.01b	0.41 ± 0.02b
T3	0.31 ± 0.020b	BDL	0.37 ± 0.14b	0.20 ± 0.02b	0.26 ± 0.03b	0.38 ± 0.01 b
T4	0.31 ± 0.02b	BDL	0.41 ± 0.02b	0.20 ± 0.00b	0.29 ± 0.02b	0.40 ± 0.03b
T5	0.33 ± 0.02b	BDL	0.51 ± 0.02b	0.19 ± 0.01b	0.30 ± 0.02b	0.46 ± 0.05b
T6	0.36 ± 0.02b	BDL	0.58 ± 0.02b	0.22 ± 0.01b	0.31 ± 0.02b	0.45 ± 0.03b

*Statistical analysis between values with different superscript letters are statistically significant ($p < 0.05$)

Table 5 - Mean values of heavy metals found in the petals

Treatment	Cu	Cd	Zn	Pb	Ni	Cr
mg kg ⁻¹						
Control	BDL	BDL	0.04±0.00	BDL	0.03±0.01	BDL
1	BDL	BDL	0.43 ± 0.01	BDL	0.11 ± 0.01	BDL
T2	BDL	BDL	0.26 ± 0.23	BDL	0.08 ± 0.26	BDL
T3	BDL	BDL	0.31 ± 0.02	BDL	0.07 ± 0.02	BDL
T4	BDL	BDL	0.28 ± 0.03	BDL	0.10 ± 0.03	BDL
T5	BDL	BDL	0.31 ± 0.02	BDL	0.06 ± 0.02	BDL
T6	BDL	BDL	0.32 ± 0.03	BDL	0.12 ± 0.03	BDL

*Statistical analysis between values with different superscript letters are statistically significant ($p < 0.05$)

Table 6 - Mean values of metals found in leaves

Treatment	Cu	Cd	Zn	Pb	Ni	Cr
mg kg ⁻¹						
Control	BDL	BDL	1.48±0.08	BDL	0.09±0.01	BDL
T1	BDL	BDL	1.82 ± 0.17	BDL	0.23 ± 0.01	BDL
T2	BDL	BDL	1.95 ± 0.21	BDL	0.21 ± 0.03	BDL
T3	BDL	BDL	2.05 ± 0.21	BDL	0.23 ± 0.02	BDL
T4	BDL	BDL	2.00 ± 0.00	BDL	0.22 ± 0.01	BDL
T5	BDL	BDL	2.10 ± 0.00	BDL	0.23 ± 0.00	BDL
T6	BDL	BDL	1.90 ± 0.28	BDL	0.23 ± 0.01	BDL

*Statistical analysis between values with different superscript letters are statistically significant ($p < 0.05$)

Table 7 - Mean values of metals found in rosebush stem

Treatment	Cu	Cd	Zn	Pb	Ni	Cr
mg kg ⁻¹						
Control	0.04	BDL	0.37 ± 0.01	BDL	0.08 ± 0.00	0.02 ± 0.00
T1	BDL	BDL	0.69 ± 0.20	BDL	0.31 ± 0.40	BDL
T2	BDL	BDL	0.79 ± 0.36	BDL	0.09 ± 0.03	BDL
T3	BDL	BDL	0.70 ± 0.29	BDL	0.07 ± 0.02	BDL
T4	BDL	BDL	1.07 ± 0.43	BDL	0.11 ± 0.06	BDL
T5	BDL	BDL	0.60 ± 0.25	BDL	0.07 ± 0.01	BDL
T6	BDL	BDL	0.73 ± 0.25	BDL	0.07 ± 0.05	BDL

*Statistical analysis between values with different superscript letters are statistically significant ($p < 0.05$)

presented the lower levels of zinc in their petals, with little variation between fertilized and those that received no topdressing. Ni concentration in petals was not statistically different in all treatments.

The leaves were those who had a higher content of zinc. It can be seen no variation comparing the two effluents used. The concentration in the leaves ranged from 1.82 mg Kg⁻¹ to 2.10 mg Kg⁻¹

Values found for Ni in the leaves were below Zn content, however were larger compared their concentration in other parts of rosebush.

The amounts of Zn in the stem were greater than those found in the petals, but lower than those found in the leaves. For Ni in the stems, the contents was low and variable among treatment and the rosebush irrigated with effluent from sand filter and received topdressing presented the highest concentration of that metal.

As demonstrated in soil, generally, roses receiving application of chemical presented the highest concentration of heavy metals.

Another study [21] evaluating the presence of potentially toxic metals in leaves and spikes of corn irrigated with treated

sanitary effluent and comparing the results with some maximum tolerance of metals in foods determined by the National Health Surveillance Agency (ANVISA) showed that the leaves of corn absorbed higher amount of metals than the spikes, exceeding the limits established by ANVISA for food. Since the work conducted, the leaves were also the tissue which absorb larger amount of metals and the petals the least amount. However, ANVISA regulates only food crops, and therefore the reuse of wastewater in agriculture recommended in cases of effluents with heavy metals content only for non food crops aimed at preserving human health.

IV. CONCLUSIONS

Although the effluent applied in rosebush did not have the standards required by law for its release in water bodies, its application in cultivated soil did not increase the heavy metal contents since the greatest impact was caused by the application of pesticides.

The pesticides application caused increase in the concentration of heavy metals both in soil and in plant.

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