

Environment

Growth and production of sunflower H-251 under the effects of the application of dilutions of water produced from petroleum

Crescimento e produção do girassol H-251 sob efeitos da aplicação de diluições de água produzida a partir de petróleo

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ABSTRACT

One of the main environmental challenges of the oil and natural gas industry is the proper disposal of large volumes of water produced during extraction. Therefore, the objective of this study was to evaluate the effects of applying dilutions of produced water on the morphometric and nutritional characteristics of sunflower. The experiment was carried out in a greenhouse set up on the west campus of the Federal Rural University of Semi-Arid. The experimental model used was randomized blocks with five treatments D0 (100% groundwater - UW and 0% produced water - TPW); D25 (75% UW and 25% TPW); D50 (50% UW and 50% TPW); D75 (25% UW and 75% TPW); and D100 (0% UW and 100% TPW) and five replicates. The sunflower was grown in 32-L pots filled with gravel (n° 0) at the base and with soil (Typical Dystrophic Red Argisol). The chemical characteristics of the aerial part (head, stem, and leaves) and roots were analyzed and subjected to analysis of variance, mean test, and orthogonal contrasts. An increase in the diameter of the stem and head (upper part of the sunflower) was observed by 77% and 65%, respectively, at their maximum values due to the application of diluted TPW. The dilutions only had an effect on Zn in the leaves and on Na in the roots.

Keywords: Environmental sustainability; Water reuse; Irrigation; Brazilian semiarid region

RESUMO

Um dos principais desafios ambientais da indústria do petróleo e do gás natural é a destinação adequada dos grandes volumes de água produzida gerados durante a extração. Assim sendo, objetivou-se avaliar

os efeitos da aplicação de diluições de água produzida nas características morfológicas e nutricionais do girassol. O experimento foi realizado em uma casa de vegetação, montada no campus oeste da Universidade Federal Rural do Semi-Árido. O modelo experimental utilizado foi em blocos casualizados com cinco tratamentos D0 (100 % de água subterrânea - UW e 0 % de água produzida tratada - TPW); D25 (75 % de UW e 25 % de TPW); D50 (50 % de UW e 50 % de TPW); D75 (25 % de UW e 75 % de TPW); e D100 (0 % de UW e 100 % de TPW) e cinco repetições. O girassol foi cultivado em vasos de 32 L, preenchidos com brita (nº 0) em sua base e com solo (Argissolo Vermelho Distrófico Típico). Foram feitas análises das características químicas da parte aérea (capítulo, caule e folhas) e das raízes, em que foram submetidos à análise de variância, teste de média e contrastes ortogonais. Foi constatado aumento do diâmetro do caule e do capítulo (parte superior do girassol) em 77 % e 65 %, respectivamente, em seus valores máximos devido a aplicação de TPW diluída. Houve efeito das diluições apenas para o Zn nas folhas e para o Na nas raízes.

Palavras-chave: Sustentabilidade ambiental; Reúso de água; Irrigação; Semiárido brasileiro

1 INTRODUCTION

Oil is one of the main current sources of fossil energy in the world (Hisham et al., 2019). In 2020, the AP volume in Brazil was 194 million m³, corresponding to a water/oil ratio of 1.13/1. In Rio Grande do Norte, in the same year, the AP volume was 57 million m³, resulting in a water/oil ratio equal to 28/1, according to the ANP database (2021). This difference between the national and state water/oil ratios is due to the more mature fields being present in the state.

However, the increase in oil production has also caused a proportional increase in the extraction of produced water from oil wells, characterized by its complex composition and a variety of contaminants highly aggressive to the environment (El Leil et al., 2021).

Therefore, the disposal of this effluent without adequate treatment can interfere with environmental sustainability. Therefore, the treatment applied should make the dangerous substances present in the effluent fit tolerable ranges for their disposal or reuse (Olajire, 2020).

On the other hand, this produced water, if applied to agricultural crops in regions affected by water scarcity, can improve the production system of crops whose

products provide raw materials for purposes other than direct human consumption (Costa, 2018), e.g., sunflower production.

Sunflower (*Helianthus annuus* L.) is a domesticated species of the family *Asteraceae*, showing high adaptability and being grown in many countries due to its variety of uses and applications, in addition to its socioeconomic importance. The oil produced from the seeds of this plant can even serve as raw material for the energy industry through its conversion into biodiesel (Simões et al., 2020). Furthermore, sunflower plants also show important agronomic features, e.g., tolerance to low and high temperatures and adaptability to different edaphoclimatic conditions.

The production of H-251 sunflower using dilutions of water produced from petroleum can contribute to both agronomic and environmental benefits. The use of this water in irrigated agriculture can promote sustainability, but the tolerance of sunflower to certain environmental stresses must be considered. The application of adequate dilutions can minimize these impacts, ensuring grain quality. In addition, research can identify safe limits for the use of produced water, preventing soil manipulation and possible toxic effects on the crop, since this exception can affect soil salinity, nutrient availability and plant physiology.

From this perspective, the present study aimed to evaluate the effects of the application of treated produced water dilutions on the morphometric and nutritional characteristics of sunflower.

2 MATERIAL AND METHODS

The study was conducted in a plant nursery located at the West Campus (Campus Oeste) of the Federal Rural University of the Semi-arid Region (UFERSA), in Mossoró-RN, located at the geographic coordinates 5°12'11,25" S and 37° 19'25,77" O, at an elevation of 13 m a.s.l. According to the Köppen classification, the climate of the region is classified as *BSh* (a dry, very hot climate with rainy summers, extending until autumn), a highly

irregular annual rainfall of 794 mm, and mean annual values of temperature and relative air humidity of 26.5 °C and 68.9 %, respectively (Alvares et al., 2013).

The volume of produced water used in the experimental tests was donated by an oil production company that acts in the Bacia Potiguar maritime region. The produced water was treated by flocculation using the organic polymer AGEFLOC DW-3753® as a flocculating agent, before being used in the fertigation of sunflower plants. In this procedure, 40 ml of the flocculant was added for each 1000 L of water. Then, these parts were mixed and rested for 48 h for segregation of the following phases: suspended material, water, and oil, according to the recommendations of Costa (2018) and Costa et al. (2020).

The dilutions of post-treated produced water were performed using well water (Açu Aquifer) managed by the Water and Sewage Company of Rio Grande do Norte (CAERN), with flow rates from 38 to 250 m³ h⁻¹, from depths between 897.0 and 1071.0 m (Oliveira, 2016).

The experimental model used in the study was in randomized blocks with five treatments D0 (100 % underground water - UW and 0 % treated produced water - TPW); D25 (75 % UW and 25 % TPW); D50 (50 % UW and 50 % TPW); D75 (25 % UW and 75 % TPW); and D100 (0 % UW and 100 % TPW) and five replications, according to the recommendation proposed by Costa (2018), aiming at mitigating the water capacity for soil infiltration, as a function of clay dispersion, and represented by the nomenclature and respective proportions of treated produced water (TPW) and underground water (UW).

The experiment employed a localized irrigation system composed of the following items: five 60-L PVC reservoirs; five self-leveling, electric circulator pumps (Metalcorte/Eberle), model EBD250076; lateral lines of 16 mm to conduct the dilutions of produced water to the pots; and microtube emitters with an average flow rate of 1.5 L h⁻¹, inserted in the lateral lines.

The crop used in the experiment was the sunflower (*Helianthus annuus*) cultivar H-251 produced by Embrapa Semiárido. The spacing used was 1.0 m between plant rows and 0.60 m between plants, with the edges of the experimental plots counting on borders. Base fertilization was performed through single applications of urea, potassium chloride, and boric acid, as recommended by Ribeiro et al. (1999), and chemical control was employed against pests and diseases. The seedlings were sown in 200-cell polyethylene trays and filled with the commercial substrate GOLDEN MIX. The seedlings were transplanted to the experimental area 12 days after sowing.

Transplanting was performed using 25 plastic pots with a capacity of 32 L, which were filled with 0.8 kg of gravel (no. zero) at the bottom, in addition to 1.0 kg of washed sand and 21 kg of soil typical from the semi-arid region of Rio Grande do Norte, classified as a Typical Dystrophic Red Ultisol, collected at the Experimental Farm Rafael Fernandes.

The experiment was carried out from September 14 to December 8, 2017, beginning after the seedlings were transplanted to the planting area. In the first 20 days of the experiment, the plants were irrigated with tap water (UW) to ensure seedling survival. After this initial period, all plants were rigorously irrigated with different proportions of treated produced water (TPW) + underground water (UW).

The gross irrigation depth for the sunflower crop (ET_c) was determined by the crop coefficient (K_c) over the reference evapotranspiration (ET_0). The reference evapotranspiration (ET_0) was determined by the Penman-Monteith equation (Allen et al., 2006). The ET_0 was estimated by using the meteorological data from the station located inside the plant nursery. The data obtained were air temperature, wind speed, relative air humidity, insolation, and solar radiation. The K_c values adopted in phases I, II, III, and IV of sunflower development were 0.52, 0.70, 0.98, and 0.81, respectively, as proposed by Cavalcante Júnior (2011).

The gross irrigation depth applied after 90 days of experiment was 435.35 mm for each dilution of treated produced water (TPW) in underground water (UW).

Characterization of the dilutions of produced water in well water. During the experiment, three samples were obtained from dilutions D0, D25, D50, D75, and D100, which were stored in 100-mL plastic containers and subsequently stored in a cooler with ice in order to maintain the temperature from 4 to 6°C and thus minimize chemical changes.

The physicochemical analyses of the dilution samples were performed in the Laboratory of Soil, Water, and Plant Analysis (LASAP) of UFERSA and comprised the following parameters: potential of hydrogen (pH); electrical conductivity (CE); calcium (Ca^{2+}), magnesium (Mg^{2+}), chloride (Cl^-), carbonate (CO_3^{2-}), and bicarbonate (HCO_3^-) expressed in $\text{mmol}_c \text{ L}^{-1}$; sodium (Na^+) and potassium (K^+) expressed in $\text{mmol}_c \text{ L}^{-1}$; copper (Cu), zinc (Zn), iron (Fe), manganese (Mn), nickel (Ni), cadmium (Cd), and lead (Pb) expressed in mg L^{-1} . All sampling, storage, preservation, and transport procedures, in addition to the physicochemical analyses of the dilution samples followed the technical recommendations of EMBRAPA (Silva, 2009) and the Standard Methods for the Examination of Water and Wastewater (Baird et al., 2017).

Table 1 shows the mean values of the physicochemical characteristics of the dilutions of treated produced water.

After sunflower harvest, performed manually 90 days after transplanting, plant development was analyzed through the followings parameters: plant height (PH), number of leaves (NL), stem diameter (SD), capitulum diameter (CD), and yield (YIELD).

At the end of the sunflower cycle (90 DAT), in addition to harvest, the plants were collected and separated into capitula, stem, root, and leaves. Then, following the technical recommendations proposed by Carmo et al. (2000) and Bataglia et al. (1983), the chemical composition of the plant tissue of sunflower was analyzed at LASAP/UFERSA. The plant parts separated were packed and identified in paper bags and subsequently dried in a forced air oven at 65 °C for 72 hours.

Table 1 – Mean values of the physicochemical characteristics of the dilutions of treated produced water

| Parameter | Unit | Dilutions | | | | |
|--------------------------------|---|-----------|-------|-------|-------|-------|
| | | D0 | D25 | D50 | D75 | D100 |
| pH | - | 8.60 | 8.73 | 8.64 | 8.46 | 8.56 |
| CE | dS m ⁻¹ | 0.56 | 0.66 | 0.66 | 0.70 | 0.71 |
| Ca ²⁺ | mmol _c L ⁻¹ | 1.39 | 1.40 | 1.20 | 1.00 | 2.00 |
| Mg ²⁺ | mmol _c L ⁻¹ | 0.11 | 0.60 | 0.80 | 0.70 | 0.50 |
| Na ⁺ | mmol _c L ⁻¹ | 2.30 | 3.80 | 3.30 | 3.00 | 4.00 |
| RAS | (mmol _c L ⁻¹) ^{0.5} | 2.65 | 3.80 | 3.30 | 3.20 | 3.60 |
| K ⁺ | mmol _c L ⁻¹ | 0.30 | 0.30 | 0.40 | 0.40 | 0.50 |
| Cl ⁻ | mmol _c L ⁻¹ | 2.40 | 4.00 | 5.00 | 3.00 | 3.00 |
| CO ₃ ⁻ | mmol _c L ⁻¹ | 0.50 | 0.60 | 0.70 | 0.70 | 0.60 |
| HCO ₃ ²⁻ | mmol _c L ⁻¹ | 2.00 | 3.50 | 4.00 | 3.00 | 4.00 |
| Cu | mg L ⁻¹ | 0.070 | 0.080 | 0.090 | 0.075 | 0.085 |
| Zn | mg L ⁻¹ | 0.027 | 0.024 | 0.028 | 0.019 | 0.054 |
| Mn | mg L ⁻¹ | 0.007 | 0.008 | 0.013 | 0.012 | 0.069 |
| Fe | mg L ⁻¹ | 0.008 | 0.035 | 0.018 | 0.022 | 0.038 |
| Ni | mg L ⁻¹ | 0.011 | 0.017 | 0.009 | 0.014 | 0.007 |
| Cd | mg L ⁻¹ | 0.006 | 0.013 | 0.008 | 0.008 | 0.012 |
| Pb | mg L ⁻¹ | 0.11 | 0.13 | 0.28 | 0.24 | 0.13 |

Source: Adapted from Costa (2018)

After drying, the plant material was ground in a mill, macerated with a mortar and pestle, and then subjected to sulfuric digestion. In this process, 0.4 g of dry plant material was put inside a digestive tube (capacity of 100 mL), which then received 2 mL of 30% H₂O₂ P.A., 4 mL of 98% H₂SO₄ P.A., and 0.7 g of a mixture of the following reagents: K₂SO₄ (100 g), CuSO₄ 5H₂O (10 g), and Se (1 g), separately (Ferreira, 2014). The concentrations of sodium (Na), iron (Fe), manganese (Mn), and zinc (Zn) were determined in the ground root and shoot samples. The sodium content was determined by flame photometry, and the other elements were determined by atomic absorption spectrophotometry.

The data of this study for the sunflower cultivar H-251 after the application of treatments were subjected to: 1) analysis of variance, by applying the F-test at 5%

significance; 2) Tukey's comparison of means at 5% probability; and 3) orthogonal contrasts for multiple comparison of means at 5% probability.

3 RESULTS AND DISCUSSION

Table 2 shows the means of the treatments for the yield (YIELD), plant height (PH), number of leaves (NL), stem diameter (SD), and capitulum diameter (CD), with their respective probabilities calculated by Tukey's test at 5%.

Table 2 – Growth and production of the sunflower cultivar H-251 irrigated with produced water dilutions in tap water

| FV | Agronomic characteristics of plants | | | | |
|---|-------------------------------------|---------------------|---------------------|---------------------|---------------------|
| | YIELD kg ha ⁻¹ | PH cm | NL uni | SD cm | CD cm |
| PFD | 0.511 ^{NS} | 0.703 ^{NS} | 0.306 ^{NS} | 0.014* | 0.062 ^{NS} |
| CV (%) | 66.37 | 18.92 | 16.45 | 28.46 | 23.23 |
| Mean | 290.53 | 58.18 | 10.68 | 0.360 | 4.384 |
| Standard error | 86.23 | 4.923 | 0.785 | 0.046 | 0.455 |
| Dilutions | Comparison of means (Tukey's test) | | | | |
| D0 | 216.34 | 52.30 | 11.40 | 0.26 a | 3.26 a |
| D25 | 409.01 | 61.70 | 11.80 | 0.28 ab | 5.36 b |
| D50 | 267.33 | 57.80 | 10.20 | 0.46 b | 4.56 ab |
| D75 | 229.29 | 58.52 | 10.40 | 0.34 ab | 4.18 ab |
| D100 | 330.70 | 60.60 | 9.60 | 0.46 b | 4.56 ab |
| Contrasts | Probability of contrasts | | | | |
| | YIELD | PH | NL | SD | CD |
| C ₁ : D100 Vs (D0+D25+D50+D75) | 0.637 ^{NS} | 0.584 ^{NS} | 0.120 ^{NS} | 0.017* | 0.654 ^{NS} |
| C ₂ : D75 Vs (D0 + D25 + D50) | 0.481 ^{NS} | 0.820 ^{NS} | 0.364 ^{NS} | 0.677 ^{NS} | 0.720 ^{NS} |
| C ₃ : D50 Vs (D0 + D25) | 0.673 ^{NS} | 0.896 ^{NS} | 0.165 ^{NS} | 0.004** | 0.660 ^{NS} |
| C ₄ : D25 Vs D0 | 0.134 ^{NS} | 0.196 ^{NS} | 0.723 ^{NS} | 0.762 ^{NS} | 0.005** |

Note: FV – Source of variation, PFD – Probability of the dilution factor, D0 – only underground water (UW), D25 – 75% UW and 25% treated produced water (TPW), D50 – 50% UW and 50% TPW, D75 – 25% UW and 75% TPW, and D100 –only TPW; C₁ = 12 m₁ + 12 m₂ + 8 m₃ +10 m₄ – 42 m₅; C₂ = 30 m₁ + 30 m₂ + 20 m₃ - 80 m₄; C₃ = 6 m₁ + 6 m₂ - 12 m₃; C₄ = 6 m₁ - 6 m₂. Means followed by the same letters in the column do not differ statistically by Tukey's test at 5% probability. ** and * significant at 1 and 5 % probability by the F-test, respectively. NS – Not significant at 5% probability by the F-test

According to the ANOVA, there was no significant effect between treatments by Tukey's test at 5% probability for yield ($p = 0.511$), as well as for none of the orthogonal contrasts. These results indicate that the TPW dilutions did not interfere with the yield of the sunflower cultivar H-251. However, there was an increase in yield in the treatments that received TPW dilutions, corroborating the study of Costa (2018), who used treated produced water to irrigate the sunflower cultivar H-360.

With regard to plant height, there was a maximum value in D25 (61.70 cm) and a minimum value in D0 (52.30 cm), indicating an increase in this parameter due to irrigation with TPW, although no statistical difference was observed between the means. It is important to note that if the efficiency of both APT dilution is determined, it is necessary to continue in order to identify the best dilution, since the necessary quantity must be preserved without damaging the soil. On the other hand, it was noted that irrigation can be carried out with smaller doses without damaging the soil and ensuring an increase in the growth of the sunflower plant.

The number of leaves showed no significant difference between treatments, indicating that the use of TPW proportions did not influence this attribute or the orthogonal contrasts. The lowest value (9.60) was observed in the treatment with the highest TPW proportion. Mello et al. (2020) found different results when growing ornamental sunflower with greywater dilutions, observing a significance in the number of leaves.

According to the ANOVA, there was a significant effect between treatments for stem diameter ($p = 0.014$) at 5 % significance. Treatment D0 differed from treatments D50 and D100, but not from the other treatments. There was a statistical difference in contrasts C1 and C3 (at 5 and 1 %, respectively), indicating that treatment D0 differs from the combination of the other treatments, and that D50 differs from the combination of D25 and D0.

Similar results were found by Melo et al. (2020), in an experiment with ornamental sunflower irrigated with greywater dilutions, in which a significant effect in

stem diameter was attributed to the use of this diluted effluent. Although the ANOVA did not reveal a significant effect for capitulum diameter, the test of means showed a significant difference between treatments. Only contrast C4 was significant (at 1% significance), which implies that dilution D25 differs from D0. Thus, capitulum diameter was influenced by the TPW dilutions. This behavior was also observed by Costa (2018) when using the same TPW dilutions in the sunflower cultivar H-360.

Therefore, the increases in plant height, stem diameter, and capitulum diameter probably occurred due to the significant potassium input into the soil, as this crop is highly demanding with regard to potassium fertilization, responding positively to K supply.

Table 3 shows the mean contents of manganese (Mn), iron (Fe), zinc (Zn), and sodium (Na) in sunflower capitula at 90 DAT. There was no significant effect between treatments for none of the elements analyzed in the ANOVA. Furthermore, there was no significant effect in the orthogonal contrasts either.

For Mn, the highest concentration occurred in treatment D25 (25.38 mg kg⁻¹), whereas the lowest (12.88 mg kg⁻¹) occurred in the treatment that only received tap water (D0). Among micronutrients, Fe was the element that showed the highest values, ranging from 63.85 (D25) to 334.75 mg dm⁻³ (D0), which was due to the higher initial content of Fe in the soil compared to the other elements studied. The maximum Zn content was observed in treatment D0 (45.90 mg dm⁻³), and the minimum in treatment D25 (30.70 mg dm⁻³).

According to Ribeiro et al. (1999), the adequate ranges of Mn, Fe, and Zn in the plant tissue of sunflower are, respectively: 300 to 600, 150 to 200, and 70 to 140 mg dm⁻³. Therefore, the Mn contents were below the adequate range in all treatments. For Fe, only treatment D0 was within the adequate range, whereas Zn showed contents lower than the adequate range in all treatments.

With a mean concentration of 1.893 g kg⁻¹ and a CV of 131.1 %, Na ranged from 0.418 to 3.474 g kg⁻¹, values observed in treatments D50 and D75 respectively. Lemos

(2016) observed high Na contents, around 0.452 g kg^{-1} , in fresh matter of forage cactus irrigated with treated domestic sewage.

The mean contents and ANOVA of the elements Mn, Fe, Zn, and Na in the stem are shown in Table 3 above, as well as the analysis of orthogonal contrasts. There was no significant difference between treatments for none of the elements. With regard to the orthogonal contrasts, the only significance was observed for Zn.

The Mn contents ranged from 5.48 to 31.78 mg dm^{-3} , corresponding to treatments D50 and D100, respectively. In a study conducted by Sedlacko et al. (2020), the authors observed an increase in the Mn content in the tissues of wheat plants irrigated with treated produced water (reduction in salinity and organic matter).

The Fe content ranged from 97.15 mg kg^{-1} (D50) to $161.38 \text{ mg kg}^{-1}$ (D100), with a mean of $126.96 \text{ mg kg}^{-1}$. Similar results were found by Costa (2019), in whose study the Fe content in cladodes of prickly pear irrigated with produced water dilutions ranged from 87.12 to $133.78 \text{ mg kg}^{-1}$.

For Zn, the maximum content occurred in treatment D100 (66.69 mg kg^{-1}), and the minimum content occurred in treatment D50 (24.89 mg kg^{-1}). With regard to the orthogonal contrasts, contrast C3 showed significance, indicating that treatment D50 differs from the combination of treatments D0 and D25. Costa (2018) reported variation in the Zn contents in the leaf tissue of sunflower irrigated with TPW dilutions, ranging from 41.43 to 46.75 mg kg^{-1} .

Overall, Zn deficiency reduces the stem elongation rate, shortens internodes, and can cause mild chlorosis, reduction, and deformation in leaves (Kerbaudy, 2019).

Table 3 – Chemical characteristics of capitula, stem diameter, leaves, and root of sunflower irrigated with produced water dilutions diluted in tap water after 90 days of experiment in the semi-arid region of Brazil, Mossoró-RN, 2022

(Continued)

| FV | Chemical concentrations | | | | | | | |
|---|--------------------------|-----------------------------|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|
| | Capitulum | | | | Stem diameter | | | |
| | Mn | Fe | Zn | Na | Mn | Fe | Zn | Na |
| | mg kg ⁻¹ | | g kg ⁻¹ | | mg kg ⁻¹ | | g kg ⁻¹ | |
| PFD | 0.761 ^{NS} | 0.323 ^{NS} | 0.254 ^{NS} | 0.490 ^{NS} | 0.499 ^{NS} | 0.932 ^{NS} | 0.117 ^{NS} | 0.619 ^{NS} |
| CV (%) | 87.03 | 111.04 | 78.51 | 131.51 | 142.68 | 94.08 | 47.26 | 87.46 |
| Mean | 19.91 | 175.53 | 37.57 | 1.893 | 27.32 | 126.96 | 52.92 | 12.15 |
| Standard error | 7.750 | 87.161 | 13.190 | 1.113 | 17.431 | 53.416 | 11.184 | 4.756 |
| Dilutions | | Comparison of means (Tukey) | | | | | | |
| D0 | 12.88 | 334.75 | 45.90 | 2.836 | 22.73 | 114.60 | 62.77 | 7.88 |
| D25 | 25.38 | 63.85 | 30.70 | 1.834 | 50.70 | 136.88 | 57.50 | 14.73 |
| D50 | 19.13 | 153.28 | 33.67 | 0.418 | 5.48 | 161.38 | 24.89 | 8.75 |
| D75 | 22.05 | 87.30 | 33.81 | 3.474 | 25.90 | 97.15 | 52.76 | 12.31 |
| D100 | 20.13 | 239.20 | 43.78 | 0.900 | 31.78 | 124.80 | 66.69 | 17.11 |
| Contrasts | Probability of contrasts | | | | | | | |
| | Mn | Fe | Zn | Na | Mn | Fe | Zn | Na |
| C ₁ : D100 Vs (D0+D25+D50+D75) | 0.714 ^{NS} | 0.252 ^{NS} | 0.693 ^{NS} | 0.672 ^{NS} | 0.857 ^{NS} | 0.988 ^{NS} | 0.248 ^{NS} | 0.273 ^{NS} |
| C ₂ : D75 Vs (D0 + D25 + D50) | 0.385 ^{NS} | 0.398 ^{NS} | 0.268 ^{NS} | 0.099 ^{NS} | 0.884 ^{NS} | 0.553 ^{NS} | 0.913 ^{NS} | 0.770 ^{NS} |
| C ₃ : D50 Vs (D0 + D25) | 0.679 ^{NS} | 0.130 ^{NS} | 0.384 ^{NS} | 0.449 ^{NS} | 0.163 ^{NS} | 0.593 ^{NS} | 0.020* | 0.667 ^{NS} |
| C ₄ : D25 Vs D0 | 0.394 ^{NS} | 0.642 ^{NS} | 0.071 ^{NS} | 0.999 ^{NS} | 0.273 ^{NS} | 0.772 ^{NS} | 0.743 ^{NS} | 0.323 ^{NS} |

Table 3 – Chemical characteristics of capitula, stem diameter, leaves, and root of sunflower irrigated with produced water dilutions diluted in tap water after 90 days of experiment in the semi-arid region of Brazil, Mossoró-RN, 2022

(Conclusion)

| FV | Chemical concentrations | | | | | | | |
|---|-----------------------------|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|
| | Leaves | | | | Root | | | |
| | Mn | Fe | Zn | Na | Mn | Fe | Zn | Na |
| | mg kg ⁻¹ | | | g kg ⁻¹ | mg kg ⁻¹ | | | g kg ⁻¹ |
| PFD | 0.412 ^{NS} | 0.653 ^{NS} | 0.047 [*] | 0.032 [*] | 0.155 ^{NS} | 0.244 ^{NS} | 0.924 ^{NS} | 0.056 ^{NS} |
| CV (%) | 96.43 | 88.58 | 41.84 | 63.60 | 47.24 | 60.08 | 49.45 | 109.46 |
| Mean | 15.60 | 291.60 | 45.34 | 4.21 | 34.05 | 657.12 | 126.58 | 18.25 |
| Standard error | 6.727 | 115.51 | 8.483 | 1.199 | 7.193 | 176.56 | 27.994 | 8.936 |
| Dilutions | Comparison of means (Tukey) | | | | | | | |
| D0 | 20.86 | 307.76 | 53.88 ^{ab} | 6.80 ^a | 41.93 | 371.68 | 130.82 | 10.95 ^{ab} |
| D25 | 25.08 | 302.28 | 64.17 ^b | 6.43 ^a | 29.90 | 475.30 | 120.40 | 13.92 ^{ab} |
| D50 | 10.54 | 160.26 | 38.68 ^{ab} | 3.20 ^a | 20.45 | 827.64 | 121.48 | 5.05 ^a |
| D75 | 10.10 | 272.74 | 25.02 ^a | 2.51 ^a | 32.40 | 777.46 | 113.08 | 17.12 ^{ab} |
| D100 | 11.42 | 414.96 | 44.93 ^{ab} | 2.14 ^a | 45.55 | 833.50 | 147.10 | 44.24 ^b |
| Contrasts | Probability of contrasts | | | | | | | |
| | 0.429 ^{NS} | 0.279 ^{NS} | 0.826 ^{NS} | 0.050 ^{NS} | 0.115 ^{NS} | 0.227 ^{NS} | 0.433 ^{NS} | 0.005 ^{**} |
| C ₁ : D100 Vs (D0+D25+D50+D75) | 0.229 ^{NS} | 0.977 ^{NS} | 0.010 [*] | 0.032 [*] | 0.967 ^{NS} | 0.234 ^{NS} | 0.728 ^{NS} | 0.537 ^{NS} |
| C ₂ : D75 Vs (D0 + D25 + D50) | 0.151 ^{NS} | 0.321 ^{NS} | 0.068 ^{NS} | 0.033 [*] | 0.098 ^{NS} | 0.080 ^{NS} | 0.906 ^{NS} | 0.510 ^{NS} |
| C ₃ : D50 Vs (D0 + D25) | 0.663 ^{NS} | 0.974 ^{NS} | 0.404 ^{NS} | 0.830 ^{NS} | 0.254 ^{NS} | 0.684 ^{NS} | 0.796 ^{NS} | 0.817 ^{NS} |
| C ₄ : D25 Vs D0 | 0.429 ^{NS} | 0.279 ^{NS} | 0.826 ^{NS} | 0.050 ^{NS} | 0.115 ^{NS} | 0.227 ^{NS} | 0.433 ^{NS} | 0.005 ^{**} |

Note: FV – source of variation, PFD – probability of the dilution factor, D0 – only underground water (UW), D25 – 75% UW and 25% treated produced water (TPW), D50 – 50% UW and 50% TPW, D75 – 25% UW and 75% TPW, and D100 –only TPW; C1 = 12 m1 + 12 m2 + 8 m3 +10 m4 – 42 m5; C2 = 30 m1 + 30 m2 + 20 m3 - 80 m4; C3 = 6 m1 + 6 m2 - 12 m3; C4 = 6 m1 - 6 m2. Means followed by the same letters in the column do not differ statistically by Tukey's test at 5% probability. ** and * Significant at 1 and 5 % probability by the F-test. NS – Not significant at 5% probability by the F-test

The highest Na contents were observed in the treatments that received the highest TPW proportion, with the minimum value occurring in D0 (7.88 g kg⁻¹) and the maximum in D100 (17.11 g kg⁻¹). Although there was no statistical difference between treatments for this element, there was an increase to the detriment of TPW dilutions. This occurred due to the high salt concentrations in the effluent, which were absorbed at considerable amounts, even with losses by leaching. Weber et al. (2017) found sodium values of 0.60, 5.13, and 0.60 g kg⁻¹ in the stem of sunflower plants irrigated with treated produced water by reverse osmosis, filtration, and tap water, respectively, in the first cycle.

The ANOVA and analysis of orthogonal contrasts for the mean values of Mn, Fe, Zn, and Na in leaves can be seen in Table 3. Only Zn and Na showed a significant difference between treatments and in at least one of the orthogonal contrasts.

There was no significant difference between treatments for Mn, with its concentrations ranging from 10.10 (D75) to 25.08 mg kg⁻¹ (D25). There was also no significant difference between orthogonal contrasts. These results are similar to those found by Costa (2018), in whose study, in experimental conditions, the sunflower cultivar H-360 was irrigated with dilutions of treated produced water, achieving a variation in the Mn content from 4.30 to 13.70 mg kg⁻¹. According to Faquin (2002), the critical threshold of this nutrient for most crops is from 10 to 20 mg kg⁻¹ in mature leaves.

The Fe concentration in the leaves ranged from 160.26 to 414.96 mg kg⁻¹, with a mean value of 291.60 mg kg⁻¹ and a CV of 88.58 %, with no significant difference between treatments or orthogonal contrasts. De Aquino et al. (2013) tested the nutritional status of sunflower cultivars irrigated with non-residual water, thus achieving a mean Fe concentration in the leaf tissue of the sunflower Hélio-251 of 100.35 mg kg⁻¹.

The Zn contents ranged from 25.02 to 64.17 mg kg⁻¹, and the mean of the treatments was 45.34 mg kg⁻¹. A significant difference was observed between treatments

($p = 0,047$). The test of means showed that D25 differed from D75 but did not differ from the other treatments. Treatments D0, D50, and D100 did not differ. With regard to the analysis of contrasts, there was significance in contrast C2, which implies that treatment D75 differs from the combination of D0, D25, and D50.

In a study carried out with ornamental sunflower varieties using fish farming effluent, Rêgo (2018) observed significance at 1% between effluent proportions in the Zn content in the leaf tissue, showing a decreasing behavior. Sandri et al. (2006) observed Zn levels ranging from 46.7 to 55.3 g kg⁻¹ in lettuce leaf tissue (a plant from the same family as sunflower) irrigated with sewage from a university.

As for Na, a maximum concentration of 6.80 g kg⁻¹ and a minimum of 2.14 g kg⁻¹ were observed, representing a mean of 4.21 g kg⁻¹ and a standard error of 1.199 g kg⁻¹, with the maximum value occurring in the control treatment (D0). According to the ANOVA, there was a significant difference between treatments ($p = 0.032$) at 5% significance. However, in the mean test, there was no difference between treatments. Contrasts C2 and C3 were significant. Therefore, D75 differs from the clustering of D50, D25, and D0, and D50 differs from the clustering of D25 and D0.

Sandri et al. (2006) found, among several elements studied, a higher concentration of Na (985, 676, and 617 mg kg⁻¹ when using sprinkler, underground drip, and surface drip irrigation systems, respectively) in the shoot part of lettuce irrigated with wastewater (university sewage treated by an anaerobic reactor), showing a statistical difference in relation to the control treatment. Costa (2019) observed lower Na values in cladodes of forage cactus irrigated with dilutions of produced water (0.298 to 0.431 g kg⁻¹). However, he observed a significant difference at 1% probability for this element by using the Tukey test.

According to the ANOVA, none of the elements analyzed in the roots showed a statistical difference at 5% significance when using the Tukey test (Table 3). However, the mean test revealed a statistical difference between treatments for Na.

The Mn concentration ranged from 20.45 (D50) to 45.55 mg kg⁻¹(D100). With regard to the contrast analysis, there was no significance in any of the contrasts. Costa (2019) found higher levels of Mn (130.48 to 177.05 mg kg⁻¹) in cactus roots irrigated with TPW dilutions. However, there was no statistical effect between dilutions either.

Although there was no significance between the Fe means, an increase in this element caused by the TPW proportions was noted, so that its minimum content occurred in treatment D0 (371.68 mg kg⁻¹) and the maximum in D100 (833. 50 mg kg⁻¹). Crisóstomo et al. (2018) found Fe levels within this range when they analyzed pineapple roots irrigated with well water (548.10 mg kg⁻¹), TPW by filtration (490.13 mg kg⁻¹), and TPW by reverse osmosis (396,27 mg kg⁻¹).

Zn concentration showed maximum and minimum values in treatments D100 (147.10 mg kg⁻¹) and D75 (113.08 mg kg⁻¹), respectively. None of the orthogonal contrasts showed a significant difference for this element. Weber et al. (2017) observed a positive effect on the concentration of Zn in the roots in the second cycle of sunflower irrigated with produced water treated by reverse osmosis (24.87 g kg⁻¹) compared to plants irrigated with aquifer water (21.77 g kg⁻¹).

The Na concentrations ranged from 5.05 to 44.24 g kg⁻¹, with the maximum value observed in treatment D100. According to the test of means, treatment D50 differed from treatment D100, not differing from the other treatments. Only contrast C1 showed a statistical difference at 1% significance, indicating that the treatment with the highest proportion of TPW differed from the combination of other treatments.

A small variation in Na (5.46 to 6.09 g kg⁻¹) was found in cactus roots irrigated with TPW dilutions in an experiment carried out by Costa (2019). Sousa et al. (2016) observed the following Na contents in the roots in the first cycle of sunflower irrigated with produced water treated by reverse osmosis and filtration and well water, respectively: 10.08, 27.99, and 22.08 g kg⁻¹.

4 CONCLUSIONS

Continuous applications of dilutions of treated produced water promoted an increase in stem and capitulum diameter by 77% and 65% considering their maximum values, respectively, also causing a greater supply of Zn in the leaves and Na in the roots. There was an increase in the variable plant height due to irrigation with APT.

Water produced from petroleum contains significant amounts of potassium, an essential nutrient for sunflowers, which respond specifically to potassium fertilization. However, its dilution is essential to avoid soil contamination.

In view of this, it is recommended that studies continue to identify the ideal dilution, ensuring soil conservation and improving sunflower development. Among the dilutions evaluated, D25 stands out (75% supply water and 25% produced water involved), which, although without statistically significant differences, favored an increase in plant height and stem and head diameters.

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