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Effects of Oilfield Water Treated by Electroflocculation and Reverse Osmosis in a Typical Brazilian Semiarid Soil

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***Abstract*—**Produced water (PW), which is water extracted along with oil, is the largest waste stream in the oil and gas industry. With the proper treatment, this wastewater can be used in agricultural irrigation. This study evaluated the effects the application of PW treated by electroflocculation (EF) and combined electroflocculation-reverse osmosis (EF-RO) on soil salinity and sodification parameters. Excessive sodium levels in PW treated by EF may affect soil structural stability and plant growth, and tends to accumulate in upper layers, displacing the nutrient K to deeper layers of the soil profile. PW treated by EF-RO did not promote salinization and soil sodification, indicating that this combined technique may be a viable alternative for oily water treatment aiming at irrigation use in semiarid regions.

***Keywords*—**Electroflocculation, irrigation, produced water, reverse osmosis, soil.

I. Introduction

AGRICULTURE is the main use of water resources in most regions of the world and is at the core of any discussion over water and food security. This activity is responsible, on average, for 70% of all global water withdrawals, and a big share of water consumption is due to crop evapotranspiration requirements. Due to population growth, urbanization, industrialization and climate change, an increase in competition for water in the future is expected, putting pressure on agriculture [1], [2]. In this respect, since water resources are becoming insufficient to satisfy demand, there has been growing interest in developing alternative sources of water for crop irrigation, mainly in arid and semiarid regions of the world [3]. Thus, the reuse of urban wastewater and industrial effluents, as well as desalinized brackish water and seawater, is being considered [4], [5].

Oilfield water, also called PW, reaches the soil surface during onshore oil exploitation and production. To extract oil from underground formations, large volumes of water are injected into oil wells [6]. PW is the largest waste stream generated in the oil and gas industries. It is a mixture of different organic and inorganic compounds. Several options are available for disposal of this wastewater, including underground injection, direct reuse if environmentally acceptable or treatment prior to disposal. Many countries are demanding more monitoring and compliance evaluations of this wastewater [6], [7].

Oil companies have also been looking for new alternatives for PW disposition and are increasingly turning to reuse for irrigation, but this wastewater contains many contaminants that can affect crops' physiological performance and nutritional quality, as well as soil physical, chemical and biological properties [4]. There are few scientific works about the impacts of this practice on the environment [5], [8] and correct irrigation management, mainly in semiarid or arid regions, where it is particularly important to avoid reduction in crop productivity and soil contamination. The inadequate reuse of treated PW in irrigation may promote nutrient losses and salt leaching if applied in excess, resulting in soil salinization and sodification [9], [10].

In recent years, several researchers have reported that EF achieved significant removals of chemical oxygen demand (COD - 94%), oil and grease (O&G - 96%), color (97%), and turbidity (99%) by PW treatment [5], [6], [11].

Reference [5] observed that the use of untreated PW blended with fresh water in higher PW concentrations tend to decrease germination speed, normal seedling percentage and seedling vigor, probably due to oil's toxic effects. On the other hand, the blending of small concentrations of PW (treated or untreated by EF) did not cause significant variations in seed germination rate and biomass production of sunflower seedlings despite the high salinity of this effluent. This plant is moderately resistant to different salinity levels in water [12], [13]. However, when comparing these results with those obtained recently by [8], who studied the effects on seed germination and early growth characteristics of sunflower after irrigation with PW treated by EF alone and combined with reverse osmosis (EF-RO), it was observed that the use of reverse osmosis membranes reduced the salinity of the effluent to acceptable levels for irrigation, and in addition, enhanced sunflower seedling vigor. RO is a membrane separation processes that can remove salts in solution, and a pretreatment like EF before RO is also recommended to remove large-diameter and free oil particles [14], [15].

The impacts of each blending ratio on soil physical and chemical properties have not yet been evaluated. These studies

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are relevant, considering that these soil phases play an important role in the soil-plant-organism interrelationship. Soil is the main carrier of water, nutrients and other elements for plant roots and soil biota [16], [17].

Soluble salts contained in irrigation water can, under certain climatic conditions, increase soil salinization and change the ionic composition of the sorption complex, modifying its physical-chemical characteristics. The increase in saline concentration decreases pH due to the increase in soil solution ionic strength, causing the displacement of Ca^{2+}, Mg^{2+} and K^{+} cations, which are important plant nutrients, from solid phase to the soil solution, reducing their availability [18], [17]. Furthermore, excessive salt concentration in irrigation water provokes reduction in water potential and reduces water absorption by seeds, which directly influences germination and plant vitality [19]-[22]. Besides this, high sodium concentrations in irrigation water affect soil structural stability and increase the infiltration rate, reducing the amount of water available to plants, and also may promote the dispersion of soil organic matter. These effects can decrease plant development, making it difficult for them to absorb nutrients [4], [16].

The present study aimed to evaluate the impacts caused by the reuse of PW treated by EF and EF-RO on physical-chemical parameters of a typical soil from the Brazilian northeastern semiarid region.

II. Materials and Methods

A. PW

The simulated PW used in this work was prepared following the procedure recommended by the Petrobras Research Center (CENPES). This PW presents equivalent parameters to real PW and for this reason is used in laboratory tests [5]: In a 2 L beaker containing 1 g of crude oil from the offshore Campos Basin (Rio de Janeiro State, Brazil), supplied by Petrobras (density 0.89 g L^{-1} and 28°API), 0.1 g L^{-1} of the emulsifiers SP-60® and TW-60® (1:1 ratio – Oxiteno S.A.), 0.9 L of distilled water and 3 g L^{-1} of synthetic sea salt – Coralife Corp.) were added. This salt concentration allowed obtaining electrical conductivity within the characteristic range (2,000-5,000 μScm^{-1}) of PWs from oil fields in Brazil's northeastern semiarid region [23]. The PW was obtained after vigorous mechanical stirring (Wigen Hauser D-500 homogenizer) at 10,000 rpm during 20 min. to form a stable emulsion.

B. PW Treatment by EF and EF-RO

Initially, the PW was treated by EF in a bench scale electrolytic reactor unit with a monopolar electrode comprised of rectangular iron plates, for a total electrode area of 700 cm^2. The alternating current (AC) supply unit had 15 V output and 60 Hz frequency, drawn from an AC/AC frequency converter (WEG model CFW 0800), connected to a step-down voltage transformer (Tecnopeltron model PLTN 100/15). All tests were conducted with a current density of 28.6 A m^{-2} for 4 min reaction time. Then the PW treated by EF was transferred to a RO system (PAM Membranes) in a feed tank and forced by a high-pressure pump into a membrane module with tangential flow, using a commercial aromatic polyamide membrane (4040-X201—TSA; Trisep Corporation) with 116.9 cm^2 area of effective permeation. This system was operated using a 1 L min^{-1} constant flow and feed pressures of 2.5 MPa [8]. Table I shows the physical-chemical analysis of the untreated synthetic PW and that treated by EF (EF-PW), according to [5], as well as the characterization of the EF-RO produced water (EF-RO-PW) according to [8].

TABLE I
PHYSICAL-CHEMICAL ANALYSIS OF UNTREATED OILFIELD PW, PW TREATED BY EF (EF-PW) AND BY EF-RO (EF-RO-PW).

Parameter	PW[a]	EF-PW[a]	EF-RO-PW[b]
pH	6.0	6.0	6.0
Conductivity (μScm^{-1})	3,583	3,448	247
TDS (mg L^{-1})	2,614	2,634	215
Turbidity (NTU)	4,780	6.6	1.0
Color ($Abs_{.400\ nm}$)	2.5	0.06	0.0
COD (mg O_2 L^{-1})	2,642	152	16
O&G(mg L^{-1})	675	21	0.0
Na (mg L^{-1})	2,158	2,022	94
K (mg L^{-1})	1,179	1,205	193
Ca (mg L^{-1})	1,301	1,236	195
Mg (mg L^{-1})	nd	nd	nd
Al (mg L^{-1})	0.40	0.30	0.1
Fe (mg L^{-1})	0.12	3.77	nd
Mn (mg L^{-1})	0.18	0.02	nd
Cr (mg L^{-1})	0.01	nd	nd
Ni(mg L^{-1})	0.05	0.04	nd
Pb(mg L^{-1})	0.11	0.09	nd

TDS: total dissolved salts; nd: not detected. [a] [5] [b] [8].

C. Effects of Irrigation with Treated PW on Semiarid Soil Properties

The soil used was an Ortic Chromic Luvisol from the municipality of Sobral (Ceará state, Brazil), which is a typical soil from Brazil's northeastern semiarid regions with onshore oil and gas exploration activity [23]. Soil samples were collected in the coordinate region 0350751 - 9582868 UTM, at a depth of 0 to 30 cm.

To accomplish the study, PVC columns (40 cm high with four segments of 10 cm and 3.0 cm diameter) were constructed. Each soil column was filled until the height of 30 cm with dried soil, disaggregated and passed through a 2 mm sieve. At the column bottom, a PVC cap with five holes of 2 mm diameter with a qualitative filter paper inside was connected to support and prevent soil loss. After soil settling, each column was placed inside a beaker, filled to 2/3 of its height with distilled water and allowed to stand for 36 hours to promote saturation. After this period, excess water was drained by gravitational action during three days to reach field capacity [24], [25].

A completely randomized factorial design was used, with two factors and three replications. The factors were water quality and depth range. The first factor had seven irrigation water qualities: distilled water (Control); EF-25 (PW treated by EF diluted to 25% v/v); EF-50 (PW treated by EF diluted

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to 50% v/v); EF-75 (PW treated by EF diluted to 75% v/v); EF-100 (PW treated by EF) and EF-RO (PW treated by EF and RO). The second factor was three soil depth ranges: 0-10 cm, 10-20 cm and 20-30 cm.

A total volume of 850 mL of each treatment water (Control, EF-25, EF-50, EF-75, EF-100 and EF-RO) was applied during four weeks at the top of the soil columns [25]. At the end of the experiment, the columns were removed and soil samples from each segment were collected, air dried and sieved through a 2 mm mesh and analyzed. Soil analysis was performed following the method described by Embrapa [26]. The particle size distribution was determined by dispersion in 1 mol L^{-1} of NaOH. Coarse sand (2-0.20 mm), fine sand (0.20-0.05 mm) and silt (0.05-0.02 mm) were separated by filtration. The clay content (< 0.002 mm) was measured in the supernatant using a Bouyoucos densimeter. The particles' density was determined by the volumetric flask method, which determines the amount of ethanol needed to complete the capacity of a volumetric flask containing dried soil. pH was measured by a potentiometer in a 1:2.5 soil/water suspension. Cation exchange capacity (CEC) was calculated from the sum of Ca and Mg, obtained from KCl 1 mol L^{-1} extraction; Na and K, obtained from Mehlich 1 extraction (HCl 0.05 mol L^{-1}+H_2SO_4 0.0125 mol L^{-1}); and Al and H, obtained from 0.5 mol L^{-1} calcium acetate extraction. Exchangeable sodium percentage (ESP), indicative of the proportion of sodium adsorbed in soil cation exchange complex, was calculated using the following equation:

$$\text{ESP} = \frac{Na}{CEC} \text{ X } 100 \quad (1)$$

where *Na* is the exchangeable sodium concentration in soil ($cmol_c/kg$) and CEC is the cation exchange capacity of soil ($cmol_c/kg$).

TABLE II
SOIL CHARACTERIZATION

Parameter	Value
Coarse sand (g kg^{-1})	394
Fine sand (g kg^{-1})	226
Silt (g kg^{-1})	220
Clay (g kg^{-1})	160
Density (g cm^{-3})	2.67
pH	6.3
Ca($cmol_c$ kg^{-1})	6.6
Mg($cmol_c$ kg^{-1})	2.2
K($cmol_c$ kg^{-1})	0.22
Na($cmol_c$ kg^{-1})	0.01
Al($cmol_c$ kg^{-1})	0.0
H ($cmol_c$ kg^{-1})	1.7
CEC[a]($cmol_c$ kg^{-1})	10.7
ESP[b] (%)	< 1
Organic C (g kg^{-1})	10.5
N (g kg^{-1})	1.1
SPE[a]:EC[d] (mS cm^{-1})	0.0
SPE:Na ($cmol_c$ kg^{-1})	0.0
SPE:K ($cmol_c$ kg^{-1})	0.0

[a] Saturated paste extract of soil

Organic carbon was measured by oxidizing the organic material with potassium dichromate in an externally heated acidic medium and titrating this with ferrous ammonium sulfate. Total nitrogen was obtained by conversion to ammonium sulfate (Kjeldahl method), oxidizing the soil with an acidic solution of $CuSO_4$ and Na_2SO_4. Electrical conductivity (EC) was obtained with a micro-processed conductivity meter (MS Mixture, model MSM 150) to read the saturated extract (SE) of saturated soil paste. Soluble Na and K contents were determined by flame photometry (Digimed; model DM 62), also reading from saturated soil extract.

Table II shows the chemical and granulometric characterization data of the studied soil.

The data were submitted to completely randomized analysis of variance (ANOVA). When significant differences were found, the Tukey test at 5% probability was applied with SAS (version 9.0). Origin (version 7.0) was used to obtain the graphs.

III. Results and Discussion

A. Semiarid Soil Characterization

A high proportion of sand fraction was observed when compared to the other textures (silt and clay) (Table II). The analyzed soil can be considered a sandy loam soil with the sand fraction predominance [27]. This fraction does not directly interfere in reactive potential of soil, which is exerted by the clay fraction together with the humified organic matter [28]. The obtained result for particles' density was consistent with the soil textural classification, since sandy-loam soils have average values of 2.6 g cm^{-3} [27].

The CEC is an indication of the soil's potential to absorb cations, indirectly representing the negative charges of the soil. With respect for this attribute, the value obtained (>10 $cmol_c$ kg^{-1}; Table II) is considered to be high but within the range typical for soils in Brazil's semiarid region [29]. This soil has moderate acidic reaction (pH = 6.3), which is an attribute of orthic chromic luvisols [27]. ESP < 15% and pH < 8.5 indicate that this soil has saline character [21], [29], [30]. Thus, it is inferred that the reuse of saline water for irrigation in this type of soil must be done wisely to avoid the salinization of the irrigated profile [4], [9], [31]. In this sense, tests were performed in soil columns to evaluate the effects of PW treated by EF (EF100) as well as blended with fresh water (EF25: with 25%v/v of EF100; EF50: with 50%v/v of EF100 and EF75: with 75% v/v of EF100) and treated PW by EF-RO (EFRO) over physical-chemical soil salinity and sodification parameters. These effects were evaluated in three different soil profile depths (0-10 cm, 10-20 cm and 20-30 cm). The occurrence of transport of ions along soil profile was also studied.

B. Effect on pH

pH values remained in the range of 6.2 to 6.9 in all depth ranges after percolations (Fig. 1) and there was no impact in original soil classification from "saline" (pH < 8.5) to "sodic" or "saline-sodic" (pH ≥ 8.5) [18], [21]. The EF-RO application

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effect in the soil pH was statistically the same, according to Tukey's test ($p < 0.05$).

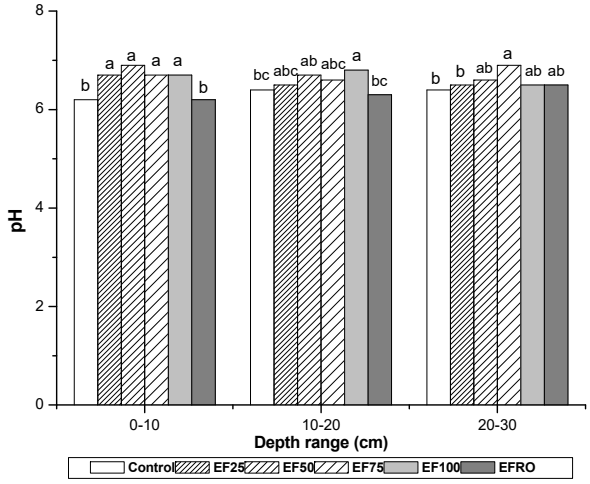


Fig. 1 Effects of PW treated by EF and EF-RO on soil pH. Average values followed by the same letter (a, b or c) in the same depth range do not differ statistically according to the Tukey test ($p < 0.05$)

The use of high salinity irrigation waters can promote a decrease in soil pH values due to soil solution gain in ionic strength. This effect causes the displacement of alkaline cations (Ca, Mg and K) from exchangeable soil sites to the soil solution [17], [30]. The results (Fig. 1) also show that there was no significant decrease in soil pH values even after application of the solution with the highest salinity (EF100).

C. Effect on Exchangeable Cations

Exchangeable cations (Na, K, Ca and Mg) from soil-exchange complex deserve special mention in evaluation of soil salinization and sodification processes [21].

The results presented in Fig. 2 (a) show that in all depth ranges evaluated, the data obtained for Ca concentrations in soil did not differ statistically after percolation according to the Tukey test ($p < 0.05$). Mg concentrations (Fig. 2 (b)) did not differ statistically in relation to the control in the topmost soil layer (0-10 cm), being close to the value obtained for the original soil (2.2 $cmol_c kg^{-1}$; Table II). The same was observed at 20-30 cm depth, a positive effect since Ca and Mg ions are essential macronutrients for plant development [32].

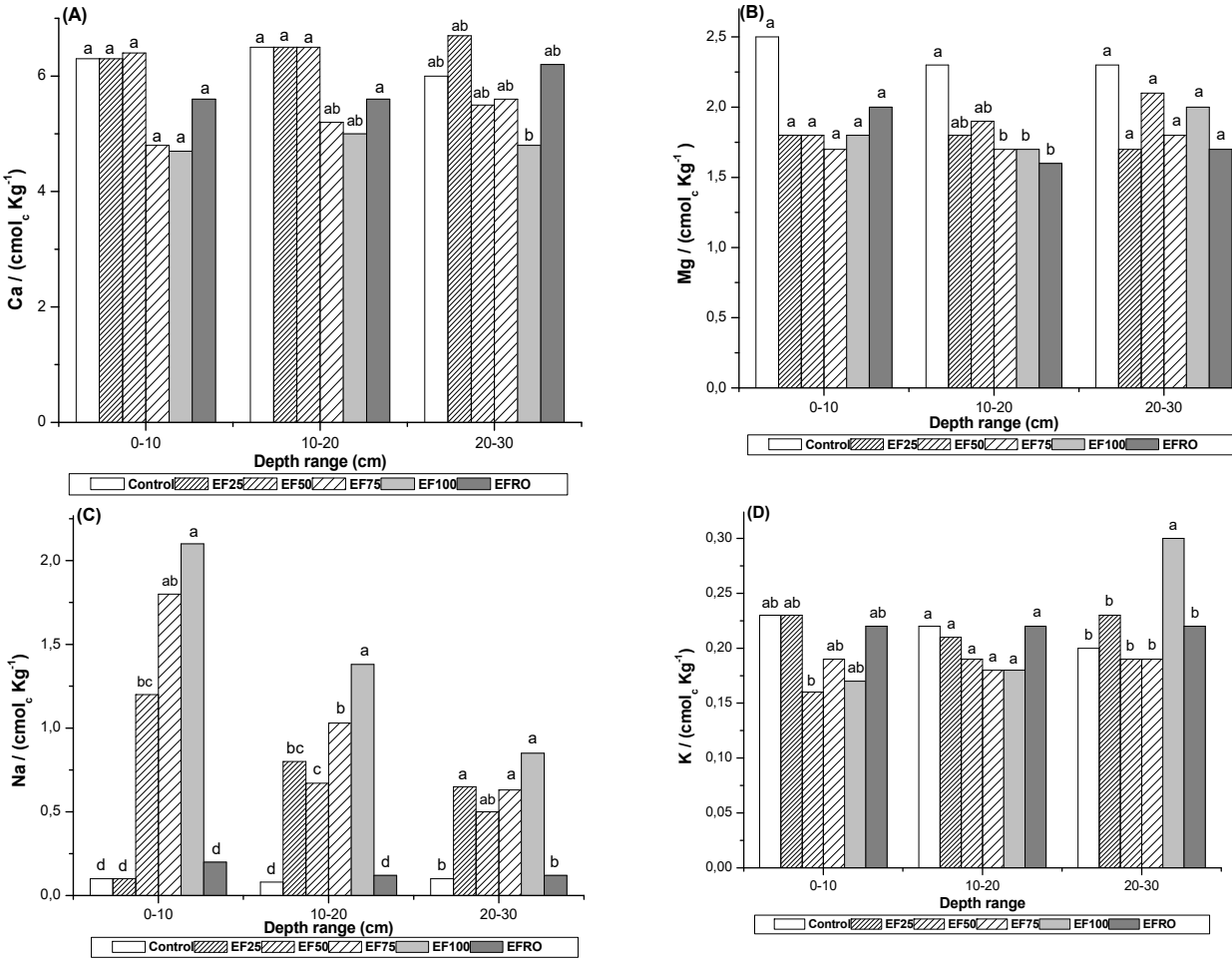


Fig. 2 Effects of PW treated by EF and EF-RO on soil exchangeable cations: (A) Ca; (B) Mg; (C) Na and (D) K. Average values followed by the same letter in the same depth range do not differ statistically according to the Tukey test ($p < 0.05$)

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At 10-20 cm depth, after the blends' percolation, the Mg value was smaller and differed statistically in relation to the control. This effect may have occurred due to the presence of monovalent ions (Na and K) at higher concentrations in these treated effluents, which induced displacement of divalent ions (such as Mg) from the solid phase to the soil solution by mass effect [18], [29].

Regarding Na, the highest concentration was obtained after EF100 effluent percolation (Fig. 2 (c)) in the surface layer (0-10 cm) and did not differ statistically, according to the Tukey test ($p < 0.05$) from EF-75 percolation. Besides this, Na concentrations after EF-RO effluent percolation also did not differ statistically from the control. The use of PW treated by EF at all dilution rates evaluated promoted considerable increases of Na concentrations in the sorption complex at all depths in comparison to the original soil (0.01 $cmolckg^{-1}$; Table II). This effect is negative since it can decrease soil productivity capacity because excessive Na concentrations tend to promote clay fraction dispersion and pore obstruction, making air and water infiltration difficult [4], [21]. Moreover, excessive Na concentration causes toxic effects in plants [13], [32].

The ANOVA showed that Na and K ions had statistically significant variations ($p < 0.05$) in each soil depth, unlike Ca and Mg ions, as a function of different effluent application. These results indicate the occurrence of transport along the soil profile for Na and K, since these ions are monovalent and have greater mobility in soil than divalent cations (Ca and Mg) [18], [29].

Fig. 3 shows the effect of depth range on average Na and K values in soil, after column percolation with PW treated effluents. This analysis aimed to evaluate transport occurrence along the soil profile.

The highest K concentration was found in 20-30 cm depth range (Fig. 3 (b)) and the highest Na concentration was found in the topmost layer (0-10 cm) (Fig. 3 (a)). These results suggest that Na ions from PW treated solutions (Table I), which are toxic to plants, tend to accumulate in the top layer (0-10 cm). This effect is not desirable, since this soil layer provides the best conditions for seed germination and emergence of seedlings [9], [13], [32]. Furthermore, the results shown in Fig. 3 also indicate that the accumulation of Na ions in upper layers moves K ions to lower soil profile layers, since K has higher mobility compared to Na [29]. This effect can be negative, since exchangeable potassium is one of the nutrients in solid phase readily mobilized to the soil solution, for uptake by plants [29]. According to [33], when K^+ availability is low, plant growth is delayed.

D.Effect on CEC and ESP

Negative surface charges in the soil colloidal fraction can attract and retain cations, such as Ca, Mg, Na, K and NH_4^+, or repel anions. This property is called CEC, which is a fundamental physicochemical parameter for soil management, since it regulates nutrient availability and soil fertility status [29].

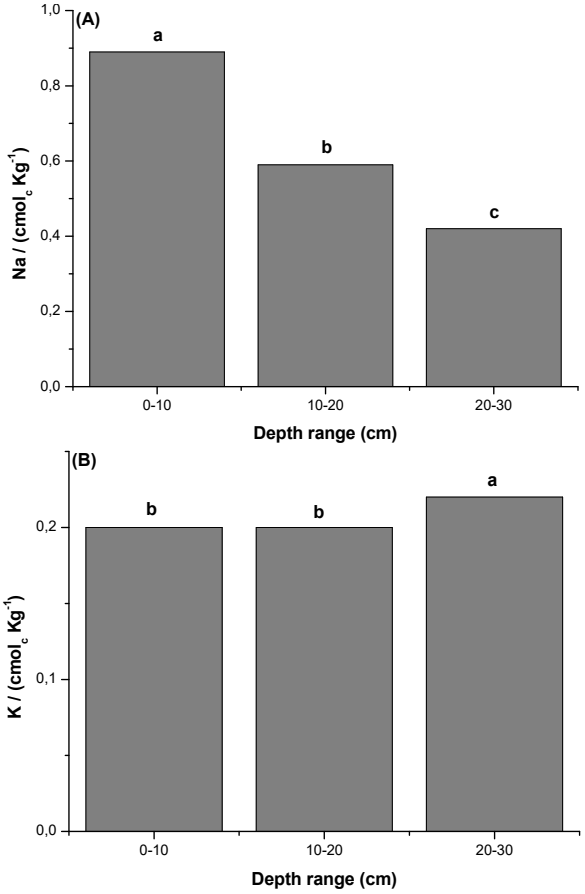


Fig. 3 Effect of depth variation on average Na (A) and K (B) values of soil after PW treated solution percolation. Average values followed by the same letter do not differ statistically according to the Tukey test ($p < 0.05$)

After percolations, CEC values did not differ statistically (Fig. 4 (a)), in all depths, according to the Tukey test ($p < 0.05$), despite the different Na values (which can promote clay fraction dispersion) and organic load levels of PW treated by EF blends and EF-RO solutions (Table I), which can change the organic soil fraction composition [18], [34]. It is also important to note that after percolations, the CEC values (Fig. 4 (a)) remained close to the soil original value (10.7; Table II).

ESP indicates Na contribution to CEC. An increase in this parameter indicates a decrease in soil infiltration capacity [29], [30]. The results shown in Fig. 4 (b) indicate that percolation with PW treated by EF and its blends promoted significant increases in ESP values of original soil (< 1%; Table II) at all depth ranges. On the other hand, the effect caused using the EF-RO effluent, which has low sodium content (Table I), did not differ statistically, according to the Tukey test ($p < 0.05$), in comparison with the control (Fig. 5 (b)).

The ANOVA results showed that ESP levels were influenced ($p < 0.05$) by depth ranges, regardless of the different characteristics of the effluents used in percolation. In this sense, Fig. 5 shows that after percolations, ESP values in soil tended to be higher in the surface layer (0-10 cm) and decrease along the soil profile, following (as expected) the

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same trend observed for Na ions in the sorption complex (Figs. 2 (c) and 3 (a)).

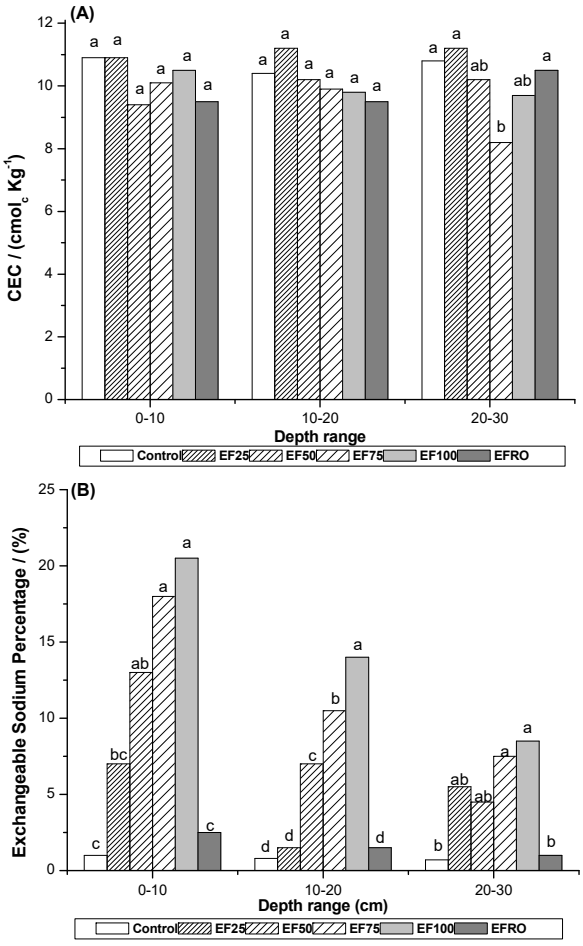


Fig. 4 Effects of PW treated by EF and EF-RO on: (A) CEC and (B) ESP of soil. Average values followed by the same letter in the same depth range do not differ statistically according to the Tukey test ($p < 0.05$)

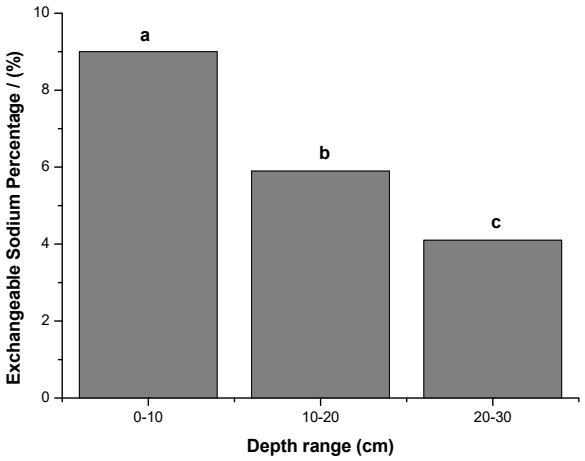


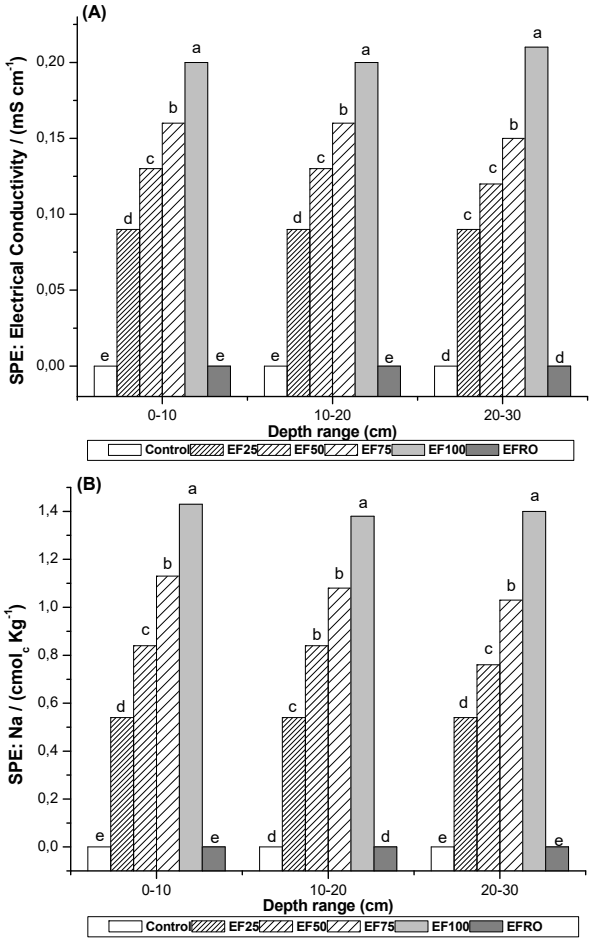
Fig. 5 Effect of depth variation on average values of ESP in soil after percolation of PW treated by EF and EF-RO. Average values followed by the same letter do not differ statistically according to the Tukey test ($p < 0.05$)

E. Effect on EC and Saturated Paste Extract

An increase in salt concentration of soil solution improves its EC. Thus, EC and soluble salts content in saturated paste extract (SPE) are parameters used to evaluate salinization and sodification grades in soil solutions [30].

As expected, the highest average values for the CE and Na in soil solutions at all depth ranges were obtained when using the EF100 effluent (Figs. 6 (a) and (b)), due to its higher salinity and sodium content (Table I).This effect is negative, since salts' accumulation in the soil solution increases the osmotic potential, hindering water absorption by plants and impairing normal development. Concerning sodium, the increase of its concentration in soil also increases the sodium adsorption ratio (SAR), which may promote clay and organic matter dispersion in the soil solid phase, reducing structural stability [4], [10], [21].

The highest average values for K in soil solution at all depths were obtained using EF100 and its blend (Fig. 6 (c)), which did not differ statistically according to the Tukey test ($p < 0.05$). On the other hand, the lowest average values for the CE, Na and K parameters in soil solution were obtained by EF-RO and control effluent, which did not differ statistically according the Tukey test ($p < 0.05$).



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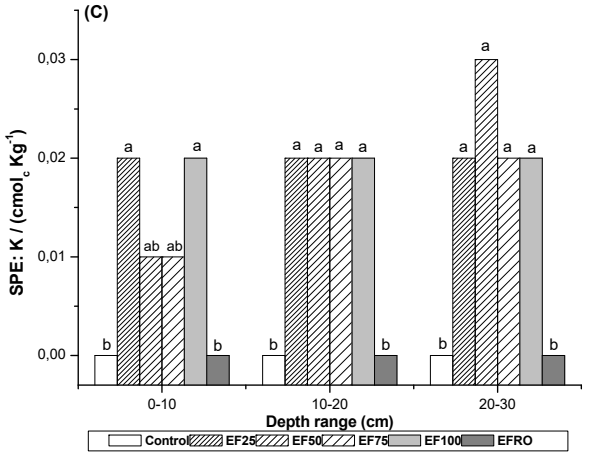


Fig. 6 Effects of PW treated by EF and EF-RO on SPE of soil: (A) EC; (B) Na; and (C) K. Average values followed by the same letter in the same depth range do not differ statistically according to the Tukey test ($p < 0.05$)

Fig. 7 shows that after percolations, K values in soil solution tended to be higher in the deepest depth range (20-30 cm). This result indicates that the use of PW treated by EF at all dilution rates tested promoted Na adsorption in the upper depth ranges, displacing the K ions towards lower layers.

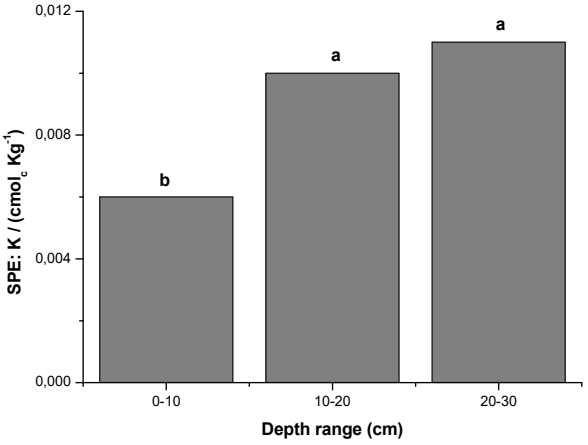


Fig. 7 Effects of depth variation on average K^+ values of SPE after percolation of PW treated by EF and EF-RO. Average values followed by the same letter do not differ statistically according to the Tukey test ($p < 0.05$)

Based on the results, the use of PW treated by EF (with or without dilution) increased salinity and sodium content in the evaluated soil to levels that can impair plant development and promote soil deterioration. Our results suggest that, in general, PW treated by EF with mean levels of salinity ≥ 497.5 mg L^{-1}, electric conductivity ≥ 977.5 µS cm^{-1}, TDS ≥ 691.5 mg L^{-1} and Na ≥ 403.5 mg L^{-1} needs extra treatment to remove excess salinity and sodium. Thus, the use of RO membranes after EF seems an interesting option for treating PW for agricultural irrigation reuse in semiarid regions like the Brazilian northeast. Furthermore, statistically speaking, the use of EF-RO treated effluent promoted the same effects as the control (distilled water) in soil and soil solution attributes.

IV. Conclusions

The use of PW treated by EF blended with fresh water promoted negative effects on solid phase and soil solution of a typical soil from the Brazilian northeast semiarid region (Ortic Chromic Luvisol), due to these effluents' salinity and sodium levels, which may influence soil structural stability and limit germination, post-seminal development and growth of irrigated crops. Excessive sodium levels in PW treated by EF effluents tended to accumulate in the upper depth ranges and moved K^+ ions to deeper layers. This effect may impair irrigated crops' development.

The effects caused to soil and soil solution attributes by using PW treated by EF-RO and the control (distilled water) were statistically similar, indicating that this combined treatment technology produces an effluent that does not promote salinization and soil sodification.

Our results indicate that the combination of EF and RO techniques has potential to be a viable alternative for oily water treatment in the oil industry, through use for agricultural irrigation in semiarid regions.

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