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Membrane Processes Treatment and Possibility of Agriculture Reuse of Textile Effluents: Study Case in Tunisia

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Abstract: Several processes have been developed to treat the textile effluents. Membrane technologies are among the most reliable processes for purifying these effluents. However, due to high costs, only reduced quantities are being treated. The recycling practices of treated textile effluents (TTE) in agriculture have not been appropriately explored. This work evaluates the quality of waters treated by membrane processes and puts forward a scenario for optimizing TTEs in agriculture. Four types of TTE have been tested to irrigate *Sesbania bispinosa* plants: water from biological treatment (BT) and water from three membrane processes after BT (Ultrafiltration (UF), Nanofiltration (NF), and Reverse Osmosis (RO)). The results indicate that the NF and RO membranes have a high affinity to remove monovalent and multivalent ions. Indeed, the removal of SO_4^{2-}, Na^+, and Cl^- by NF was 83, 61, and 55%, respectively. Thus, the RO reduces approximately 96% of these elements. Irrigation with NF and RO waters has no negative effect on the soil and *Sesbania* plants, contrary to BT and UF waters. It appears that the reuse of TTE resulting from BT is not a good alternative; however, by carrying out additional treatments by NF and RO, their reuses have been made possible. The achieved results are a proposal to simultaneously solve three major problems affecting most of the world's population: (1) environmental pollution by reducing the discharge of untreated textile effluents and improving the quality of this discharged water; (2) the pressure on water resources in the agricultural sector by replacing a conventional resource with a non-conventional resource (TTE); and (3) the lack of fodder, especially in the summer, by opting for crops that adapt to the quality of these TTE.

Keywords: treated textile effluents; membrane treatments; water reuse in irrigation; non-conventional waters; water characterization; soil; *Sesbania bispinosa*

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1. Introduction

The textile industry generates large volumes of contaminated wastewater [1,2] that constitute a source of pollution of surface water and groundwater [3,4]. Consequently, environmental legislation requires that textile factories should treat these effluents before their discharge. Generally, industrial on-site water treatment plans carry out a primary or physico-chemical pre-treatment [5] followed by biological treatments that reduce dissolved pollutants (mainly organic matter). However, these traditional decontamination systems do not seem sufficient, especially for agricultural reuse.

Several enhanced purification processes have been developed in the industrial sector to further eliminate pollutants such as chemical oxidation, electrolysis, biodegradation, adsorption, chemical coagulation, and membrane processes [6–10]. Among them, membrane processes have shown interesting results [11–13]. In terms of pore sizes, there are four types of membranes; microfiltration (MF) (0.05–1.0 μm), ultrafiltration (UF) (0.005–0.5 μm), nanofiltration (NF) (0.0005–0.01 μm), and reverse osmosis (RO) (0.0001–0.001 μm [14].

MF membranes can be used for versatile fields needing separation of bacteria, aerosols, viruses, and innumerable macromolecules from fluids [15]. They are used in wastewater treatment; as a pretreatment filter before UF, NF, and RO membrane-based processes; for separation of macromolecules in pharmaceutical industry; and also in textile effluents treatment [15,16]. Indeed, literature indicates that ceramic MF membranes showed a considerable removal efficiency to BOD (39%), sulfates (34%), chlorides (33%), TDS (31%), color (26%), COD (25%), and turbidity (21%) from textile effluents treatment study [16]. The disadvantage of this process is that the expected degree of purification in MF is lower than in other membrane processes [15].

UF process is typically used to remove viruses, emulsified oils, metal hydroxides, colloids, proteins, and other high molecular weight materials from water and other solutions [17]. It operates at low transmembrane pressure to remove dissolved substances, and its membranes are capable of handling high flux [18]. Furthermore, Barredo-Damas et al. [19] deduced that ceramic UF membranes can be used as an alternative pretreatment to adapt textile effluent for a later stage of NF and RO.

The combination of UF and RO is known as NF, which combines a high removal rate with low energy consumption [20]. It is a very selective process for separating salts, sugars, or dye molecules [21]. Thus, Aouni et al. [22] deduced that the NF process is effective in reducing conductivity, COD, and color in industrial effluents. Indeed, a study that deals with the performance of a certain NF membrane (NF-270-2540 FILMTEC NF) showed that this process effectively reduces dyes, COD, and organic carbon [23].

RO is a method that removes various species and is widely used in the desalination process [20]. Due to the fact that RO membranes are prone to scaling and fouling, RO is rarely a stand-alone treatment process [24]. RO processes can simultaneously remove hardness, color, many types of bacteria and viruses, and organic contaminants such as agricultural chemicals [25].

In addition to the harmful effects of untreated textile effluents on the environment, there is also the problem of water scarcity [26], the high demand for water in the agricultural sector [27], and the lack of fodder in some countries. This implies that the reuse of these treated waters in agriculture, by adopting plants to the quality of these waters, can be a form of recycling water and nutrients. Their environmental impacts can be reduced [28]. Another problem that arises in the agricultural sector is the use of chemical fertilizers, especially nitrogenous fertilizers that lead to the eutrophication of surface and groundwaters and the degradation of the quality of the environment [29–31]. This has drawn attention towards the use of nitrogen-fixing plants in order to increase soil fertility. This is the case of *Sesbania bispinosa* (Fabaceae family).

Many studies have focused on wastewater depollution processes and their characterization for reuse in agriculture [32]. In a circular economy approach, some authors have assessed the possibility of reusing treated textile effluents as an influent in textile dyeing processes. Some researchers have studied the problems of soil salinization, whereas others have been interested in the behavior of metallic trace elements (MTEs) present in these waters with respect to irrigated soils as well as organic matter [33,34] and enzymatic activity as a bioindicator of the quality of irrigated soils [35]. However, few studies have been carried out on the agricultural valorization of textile effluents subjected to different membrane treatments. It is within this context that our work exists, which aimed to propose effective systems for depolluting textile effluents and to evaluate the possibility of reusing them in agriculture. This was carried out through (i) the characterization of wastewater from a textile dyeing plant subjected to biological treatment (BT) and complementary treatments by UF, NF, and RO; (ii) the determination of the pysico-chemical modifications of the soil induced by these treated effluents and; (iii) the evaluation of their effects on the "*Sesbania bispinosa*" plant.

2. Materials and Methods

2.1. Soil Sampling Site

The soil for the microcosm study was taken from the layer 0–30 cm of agricultural land of Ksar Hellal region (Monastir Governorate, Tunisia). This region is under semi-arid climate with an average annual rainfall of 300 mm. The samples were taken at random from three points. The soil distribution size according to the Robinson method shows a sandy loamy texture with 63.2% of sand, 17.3% of clay, and 16.1% of silt.

2.2. Textile Effluent Treatment

The textile effluents were available from the sewage treatment plant of the Sitex Textile Company, located close to the soil-sampling site in Ksar Hellal. This company specialized in the production of Denim fabrics and uses the indigo blue dye ($C_{16}H_{10}N_2O_2$) in the production processes. It is a dye insoluble in water and poorly soluble in solvents [8]. The sewage treatment plant performs only the biological treatment (BT) of textile effluents by activated sludge. Membrane treatment units have been set up on a pilot in the industry's wastewater treatment plant to ensure the additional treatment of BT water by Ultrafiltration (UF), Nanofiltration (NF), and Reverse Osmosis (RO). The membrane characteristics are shown in Table 1 below:

Table 1. Membrane characteristics.

Parameters	UF Membrane [8]	NF Membrane NF 270 [36,37]	RO Membrane BW30 [36,37]
Material	TiO_2	Polyamide	Polyamide
Membrane area (m^2)	0.155	2.5	2.5
Molecular weight cut-off (MWCO)	150 kDa	200–400 Da	>100 Da
Manufacturer	Dow Filmtec, Santa Ana, CA, USA	Dow Filmtec, Santa Ana, CA, USA	Dow Filmtec, Santa Ana, CA, USA
Salt rejection (%)			99.4
NaCl rejection (%)	-	50	-
$MgSO_4$ rejection (%)		98	-
Pure water permeability (L/m^2 h. bar)	220 ± 15	7.6 ± 0.2	3.2 ± 0.2

2.3. Water Irrigation

Four qualities of TTE were used for irrigation: (i) biological treatment water (BT), (ii) Ultrafiltration (UF), (iii) Nanofiltration (NF), and (iv) Reverse Osmosis (RO). In addition, well water (W) pumped from a 45-m-deep aquifer was used as a control.

2.4. Experimental Setup

The experiment was carried out in 2019 at the National Institute for Research in Rural Engineering, Water, and Forests (INRGREF). The chosen device was in total randomization. The plant material used was *Sesbania bispinosa*. *Sesbania* seeds were planted in pots filled with peat and watered with drinking water. Seed emergence took place on 27 March 2019. At the five to six leaves stage, the seedlings were transferred to plastic pots (30 cm: diameter × 32 cm: height) loaded with 15 kg of soil at the rate of two seedlings per pot. The pots were aligned in five rows with five repetitions per water quality for a total of 25 pots. A cycle of 25 irrigations, for each quality of water, was carried out with a dose set at 28.6 mm for each pot. Since the plants' transfer to the pots (13 May 2019), the diameter and length of the stems were regularly monitored. At the flowering stage (2 September 2019), the plants were collected. The harvest consisted of manually extracting each plant from the soil, with the entire root system. The roots were washed with distilled water. Biomasses were determined by weighing fresh and dry matter from leaves, stems, and roots. The dry matter was determined after placing the fresh matter in the oven at 60 °C until the stabilization of the dry weight.

After grinding the dry matter of the plants, the different organs were analyzed for their contents in potassium (K^+), sodium (Na^+), and metallic trace elements (Cr, Co, Cu, and Ni). K^+ and Na^+ were determined by flame spectrophotometry (Jenway, PFP7, France) and MTEs by atomic absorption spectrophotometry (Perkin Elmer, AAnalyst 400 AA Spectrometer; WinLab32 Software).

2.5. Soil Sampling and Analysis

The soil characteristics were determined before and at the end of the irrigation cycle. At the end of the experiment, soil samples were taken at several heights from each pot. The samples were taken manually using plastic gloves, air-dried, and passed through a 2 mm sieve. Soil samples were homogenized before.

pH was determined using a digital pH meter (XS instruments, pH 70+ DHS, Italy) from a soil-water mixture 1/2.5. Electrical conductivity and mineral elements were determined from the soil solution extracted from the saturated soil paste (EC_e) according to the US Salinity Laboratory Staff [38] by conductimetry (AZ instruments-8361-Cond. & TDS Pen, Taiwan). K^+ and Na^+ soil contents were determined by flame spectrophotometry (Jenway, PFP7). The Ca^{2+}, Mg^{2+}, Cl^-, and HCO_3^- ions were assayed by titrimetry; SO_4^{2-} ions were assayed by nephelometry using a UV-Visible spectrophotometer (Jenway 6305, France). MTE contents were determined by atomic absorption spectrophotometry [39]. The choice of analyzed MTEs was based on the Tunisian standard NT 106.02 for textile industry's treated wastewater discharges.

2.6. Water Analysis

Treated textile effluents and well water were sampled in polyethylene bottles at three different dates and analyzed for the pH, electrical conductivity (EC_w), mineral composition (K^+, Na^+, Ca^{2+}, Mg^{2+}, Cl^-, SO_4^{2-} and HCO_3^-), and MTEs contents.

2.7. Statistical Analysis

At least three replicates were performed for each data, and the means were compared by Tukey's test at the confidence level of 0.05 using SPSS software (IBM SPSS statistics, v20). The different letters presented in the figures and tables indicate significant differences at $p < 0.05$. The bars indicate the standard deviation (SD).

3. Results and Discussion

3.1. Physico-Chemical Quality of Treated Textile Effluent (TTE)

3.1.1. Biological Treatment

The effluents leaving the textile dyeing plant after BT do not comply with Tunisian Standards NT 106.02 for discharge water and NT 106.03 for reuse in agriculture. This is due to exceeding values of pH (8.6), EC (8.1 dS/m), and Cl^- (28.5 me/L) and SO_4^{2-} (27.1 me/L) ions contents (Table 2) related to the use of sodium dithionite ($Na_2S_2O_4$). This reducing agent is used to make indigo blue soluble in water, whose pH becomes alkaline, with pHs varying between 8.0 and 9.5. Similarly, Chockalingam et al. [40] report that an effluent sample collected from the city of Salem in Tamilnadu, India, contains a blue dye with a pH of 8.6. Vigneshpriya and Shanthi [41] also mentioned a pH of 9.5 for the effluent collected from Karur, a textile town in Tamilnadu.

3.1.2. Membrane Treatments

By carrying out additional treatments to BT effluents, we observed an improvement in their qualities. In fact, the pH remained basic after UF and NF (7.9–8.4), and became neutral with RO (7.1) (Table 2). These pH values are consistent with the two Tunisian standards NT 106.02 (6.5–9.0) and NT 106.03 (6.5–8.5).

Textile wastewater has high conductivity due to the use of various salts during the dyeing process [42–45]. This is the case in our study, where we found that the BT effluents exhibit a high electrical conductivity (8.1 dS/m) that exceeds the Tunisian Standards NT 106.03 and NT 106.02. Additional treatments with UF, NF, and RO have contributed to a significant reduction ($p < 0.05$) in the salinity of water to 7.01, 2.62, and 0.20 dS/m, respectively. A slight reduction was observed following UF treatment. Özgün et al. [46] have shown that this type of treatment can be considered as a pre-treatment method before proceeding to NF. The NF treatment made it possible to reduce the amount of salts contained in the BT water to three times less, whereas with the RO we managed to desalinate the textile effluents. Therefore, it can be inferred that nanofiltration (NF) plays an important role in reducing the salinity levels of textile effluents, as demonstrated by Aouni et al. [22]. It is important to note that the NF treatment process is used in pre-treatment unit operations in seawater desalination processes [47,48]. Regarding RO, this treatment process effectively eliminates salinity in biologically treated textile effluents. Thus, the higher salinity reduction efficiency for the RO membrane is due to its relatively denser selective surface layer compared to the NF membrane [49]. The mineral ion contents strongly increase after BT compared to the well water (Table 2), but significantly decrease after adding NF and RO, with values complying with the Tunisian Standards. The lowest eliminations of chemical elements were observed with UF treatment. The removal of SO_4^{2-}, Na^+, and Cl^- were, respectively, 13, 10, and 12%. The NF shows an efficiency to reduce these elements; indeed, SO_4^{2-} was reduced by 83% and Na^+ and Cl^- were reduced, respectively, by 61% and 55%. Several researchers note that NF is characterized by a high rejection coefficient of multivalent ions (up to 90% for sulfate ions) compared to monovalent ions such as Na^+ and Cl^- (less than 60%) [50,51].

Table 2. Characterization of the well water (W) and the TTE. The average values are compared to the Tunisian standards. W: Well water; BT: Biological treatment; UF: Ultrafiltration; NF: Nanofiltration; RO: Reverse Osmosis; and ECw: electrical conductivity of irrigation water. ND: Not determined.

			Complementary Treatments			Tunisian Standards	
	W	**BT**	**UF**	**NF**	**RO**	**NT 106.02**	**106.03**
Cations							
pH	7.2 ± 0.14 a *	8.6 ± 0.17 a	7.9 ± 1.80 a	8.4 ± 0.46 a	7.1± 1.04 a	6.5–9	6.5–8.5
Ecw (dS/m)	2.20 ± 0.12 b	8.13 ± 0.63 d	7.01 ± 0.37 c	2.62 ± 0.20 b	0.20 ± 0.03 a	5	7
Ca^{2+} (me/L)	8.90 ± 1.29 b	11.80 ± 1,82 b	8.60 ± 4.26 b	2.10 ± 0.22 a	ND		
Mg^{2+} (me/L)	6.20 ± 0.27 b	15.30 ± 0.97 d	12.00 ± 1.70 c	4.40 ± 1.11 b	ND		
K^+ (me/L)	0.13 ± 0.76 a	3.33 ± 0.53 b	3.07 ± 0.13 b	0.30 ± 0.34 a	0.12 ± 0.06 a		
Na^+ (me/L)	10.48 ± 2.25 b	51.78 ± 4.88 d	46.50 ± 3.68 d	20.31 ± 0.41 c	2.00 ± 0.44 a		
Anions							
Cl^- (me/L)	11.56 ± 0.63 b	28.53 ± 4.88 c	25.22 ± 4.74 c	12.74 ± 1.70 b	0.81 ± 0.10 a	19.75 (700 mg)	56.41 (2000 mg)
HCO_3^- (me/L)	4.50 ± 2.20 b	24.90 ± 1.60 e	21.10 ± 1.52 d	8.80 ± 1.64 c	0.50 ± 0.50 a		
SO_4^{2-} (me/L)	8.41 ± 1.99 b	27.14 ± 3.37 c	23.57 ± 2.85 c	4.72 ± 1.31 ab	1.12 ± 0.59 a	10.41 (500 mg)	
SAR	2.82 ± 0.75 a	7.68 ± 0.58 b	9.56 ± 2.68 bc	12.82 ± 2.56 c	ND		
Cd (mg/L)	ND	0.08 ± 0.01 b	0.08 ± 0.01 b	0.01 ± 0.01 a	ND		0.01
Co (mg/L)	ND	0.62 ± 0.12 b	0.50 ± 0.12 b	0.06 ± 0.07 a	0.01 ± 0.01 a		0.1
Cr (mg/L)	0.01 ± 0.00 a	0.21 ± 0.04 b	0.24 ± 0.05 b	0.04 ± 0.01 a	0.02 ± 0.01 a	0.5	0.1
Ni (mg/L)	ND	0.74 ± 0.02 c	0.63 ± 0.04 b	0.06 ± 0.05 a	0.02 ± 0.01 a	1	0.2

* The means of five observations (±SD) followed by the different letters in each line are significantly different according to Tukey's test at the level of $p < 0.05$.

Knowing that RO is a water desalination technique, we assumed that the BT water subjected to RO complies with the standards regarding major ions. Indeed, Na^+ and Cl^- were eliminated by 96% and 97%. Thus, 96% of the sulfates in the biological treatment waters were rejected.

These results indicate that the NF and RO membrane methods can be considered as promising technologies that can be used for the treatment of textile effluents. The reason for the greater use of membrane filtration systems in the textile sector is mainly due to, for example, the membrane's ability to remove pathogenic microorganisms and salts as well as to control the disinfection by-products (DBP) precursor [6,22]. Other treatment processes have proven to be more efficient for the removal of dyes such as ozonation [52] and adsorption [53,54]. According to Mohammad Ali et al. [53], the use of Fe_3O_4-SiO_2

nanoparticles as an adsorbent material leads to the elimination of 99% of the methyl blue dye from aqueous solutions. Although both processes have their own advantages, there are also disadvantages associated with their uses. Membrane fouling is the main limitation of wastewater filtration which limits the performance of membranes [24,55], whereas the application of adsorption methods can be limited by the inconvenience of regeneration and the high price when using activated carbon [56]. Consequently, several studies have proposed to obtain a hybrid treatment process combining membrane techniques and adsorption with cost/performance advantages [57,58].

The sodium risk of irrigation water is generally expressed by the sodium adsorption ratio (SAR). This index quantifies the proportion of Na^{+} ions in relation to Ca^{2+} and Mg^{2+} ions in the water. SAR values for irrigation water ranged from 2.8 for well water to 12.8 for nanofiltered water. The SAR for RO water was not determined because Ca^{2+} and Mg^{2+} ions were not detected. Several researchers mention that high SAR values indicate a substitution of Ca^{2+} or Mg^{2+} for Na^{+} in the soil through the cation exchange process, which causes a reduction in soil permeability and restricts the circulation of air and water through the soil system, thus leading to the development of alkaline soil [59–61]. This is not the case in our study, where we deduce according to the classification of Richards (1954) based on the values of SAR [59] that all water qualities belong to the category " excellent", except for NF water, which belongs to the "good" category. Accordingly, this explains how TTE can be used in irrigation without risk to the soil.

MTEs' contents exceed those set by the Tunisian Standards except for NF and RO.

Therefore, we deduce that the biological pre-treatment as carried out in our case study partially improves the water quality, and the water resulting from this treatment remains highly polluting. A complementary approach is essential for healthy use. Membrane treatments of textile effluents by NF and RO allow, to different degrees, improvements in quality, suggesting either the recycling of water in the factory's production line, or agricultural recovery. Nevertheless, these techniques are energy-intensive, resulting in high costs, and produce highly concentrated water discharges. Therefore, other options are to be considered in synergy with these techniques or separately. Agricultural recovery seems to be the unavoidable option but comes up against the high salinity of water from biological treatment, which exceeds the salinity tolerance of most plants and affects the properties of soils irrigated by these waters. Softening by coupling with well water is a simple and inexpensive solution.

3.2. Impact of Irrigation by TTE on Soil Quality

The soil before irrigation (IS) exhibits a fairly neutral pH (7.3). After irrigation by BT, UF, and NF waters, soil pH significantly increases ($p < 0.05$), however, it remains near neutral with RO waters (Table 3). Such alkaline waters may reinforce the soil sodicity condition and have adverse effects on microflora and crop growth [62,63].

Soils irrigated by BT waters showed a dominance in Na^{+} and Cl^{-} ions compared to control soil where Ca^{2+} and Cl^{-} slightly dominate, whereas no significant differences were noticed with UF and NF waters; Cl^{-} concentration significantly decreased ($p < 0.05$) after RO water. However, Na^{+} is one of the most undesirable elements in soil. This element decreases the permeability of the soil, which reduces the water available for the plants and can affect their germination [64]. Awasthi et al. [65] showed that calcium and magnesium are low in sodium soil. It is the opposite in our soils except when using RO water that is demineralized.

The highest ECe in soil irrigated by BT are in correlation with the high concentrations of Na^{+} and Cl^{-} ions in these soils (ECe = 11.5 dS/m). Lower ECe values were recorded for the less salinized soils irrigated by UF, NF, and RO (8.3, 6.6, and 1.5, respectively).

The results of MTEs analyses (Cd, Co, Cr, and Ni) in the soil treated by the different qualities of water are presented in Table 3. The concentrations do not vary very significantly depending on the irrigation water. Indeed, despite the concentrations that exceed the Tunisian standard NT 106.3 in BT and UF waters, the MTEs contents in the soil are well below the range of values set by Kabata-Pendias and Pendias [64] and the maximum tolerable fixed by the WHO [66].

Compared to the soil in its initial state (IS: before irrigation), irrigation with RO water causes a reduction in MTE levels. This suggests that these elements are leached by drainage water or absorbed by plants. This leaching depends on several factors such as soil pH and texture [67]. Thus, Kabata-Pendias and Pendias [64] mentioned that the pH and the total concentrations of some MTEs such as Cd and Ni are very close. Indeed, the solubility of metals is high in acid soils [64,68]. Under more alkaline conditions, as is the case in our study (7.3 < pH > 8.6), the solubility of some MTEs is not correctly predicted by pH [69] because under these conditions, pH is most often coupled with other physico-chemical characteristics such as organic carbon content and soil texture [69,70].

On the other hand, an increase in MTE contents in the soil was observed in the case of irrigation by BT waters with a lesser extent in the case of UF and NF waters. This reflects a phenomenon of accumulation of MTEs in the soil while remaining within the standards. The accumulation effect of MTEs should be studied in the long term to assess the impact of these waters on the quality of the soil since several studies have deduced that the long-term use of wastewater could affect the quality of the soil through MTEs pollution and increased salinity [71,72].

Thus, it has been shown that the content of MTEs, essentially Cr, in the surface layer of the soil is known to increase due to contamination from various sources, the main ones being industrial wastes such as Cr pigments and tannery waste and leather manufacturing waste [64].

Table 3. Effects of irrigation by TTE on the soil. IS: Initial status; W: Well water; BT: Biological treatment; UF: Ultrafiltration; NF: Nanofiltration; and RO: Reverse Osmosis.

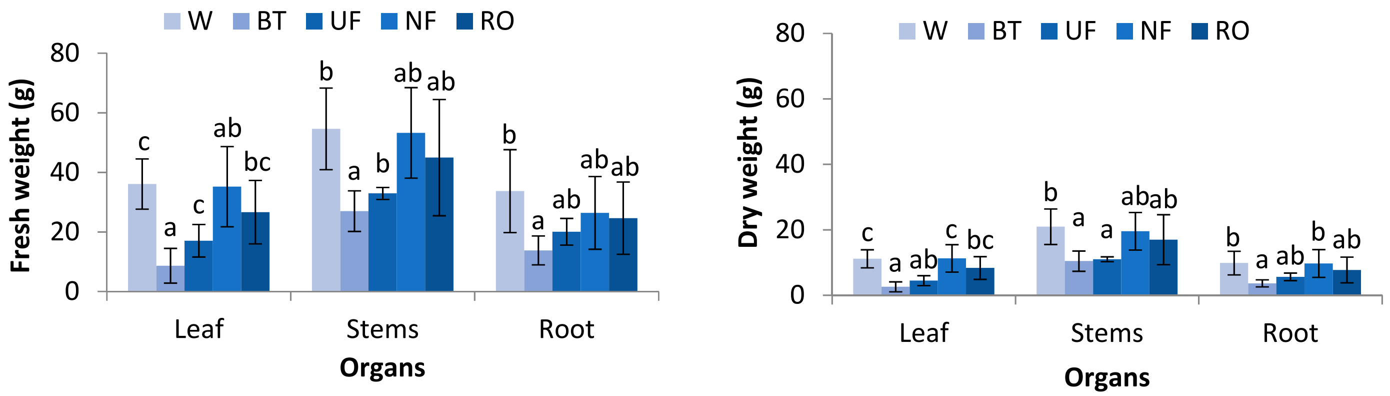
			Complementary Treatments				Ranges and Maximum Tolerable of ETM in the Soil (mg/kg)	
	IS	W	BT	UF	NF	RO	[64]	[66]
pH	7.4 ± 0.13 a *	7.5 ± 0.29 a	8.7 ± 0.19 c	8.2 ± 0.11 b	8.3 ± 0.08 bc	7.3 ± 0.07 a		
ECe (dS/m)	1.21 ± 0.07 a	5.24 ± 0.03 b	10.90 ± 0.71 e	8.31 ± 0.30 d	6.66 ± 0.26 c	1.53 ± 0.20 a		
Cations								
Ca^{2+} (me/L)	5.60 ± 0.65 a	13.00 ± 2.12 a	39.63 ± 4.42 b	30.10 ± 9.38 b	15.50 ± 6.01 a	5.50 ± 1.00 a		
Mg^{2+} (me/L)	3.7 ± 0.27 a	9.50 ± 1.68 b	14.88 ± 2.87 b	12.00 ± 4.47 b	9.50 ± 2.52 b	2.00 ± 0.50 a		
K^{+} (me/L)	0.34 ± 0.01 ab	0.49 ± 0.11 bc	0.59 ± 0.08 c	0,50 ± 0.11 bc	0.50 ± 0,13 bc	0.20 ± 0.02 a		
Na^{+} (me/L)	4.44 ± 0.27 a	30.24 ± 1.97 b	52.02 ± 0.95 d	39.78 ± 6.67 bc	41.71 ± 9.21 cd	7.95 ± 0.53 a		
Anions								
Cl^{-} (me/L)	6.77 ± 1.18 a	33.13 ± 1.43 b	54.68 ± 15.43 c	40.63 ± 3.62 b	33.54 ± 7.21 b	6.77 ± 1.20 a		
HCO_3^{-} (me/L)	2.50 ± 0.00 a	4.37 ± 0.48 ab	11.50 ± 1.78 c	7.60 ± 2.86 b	3.80 ± 0.27 a	3.83 ± 1.26 a		
SO_4^{2-} (me/L)	2.25 ± 0.67 a	15.66 ± 1.36 a	43.37 ± 13.47 b	35.25 ± 5.28 b	30.65 ± 6.30 b	4.36 ± 1.31 a		
MTE								
Cd (mg/kg)	0.66 ± 0.38 abc	0.76 ± 0.09 bc	0.96 ± 0.02 c	0.86 ± 0.04 bc	0.55 ± 0.09 ab	0.37± 0.04 a	0.08–1.61	4
Co (mg/kg)	0.89 ± 0.50 a	1.64 ± 0.42 b	1.77 ± 0.31 b	1.71 ± 0.15 b	1.36 ± 0.17 ab	1.02 ± 0.15 a	3–58	-
Cr (mg/kg)	4.12 ± 0.61 bc	3.47 ± 0.28 ab	5.21 ± 0.26 d	4.26 ± 0.31 c	4.21 ± 0.26 c	3.16 ± 0.28 a	4–1100	-
Ni (mg/kg)	2.83 ± 0.83 ab	2.40 ± 0.76 ab	3.50 ± 0.22 c	2.79 ± 0.26 ab	2.56 ± 0.19 ab	1.96 ± 0.97 a	3–110	107

* The means of five observations (±standard deviation) followed by the different letters in each line are significantly different according to Tukey's test at the level of $p < 0.05$.

3.3. Impact of TTE on Sesbania Plants

3.3.1. Effects on Biomass

One of the effects of irrigation on the plant is a significant decrease ($p < 0.05$) in the yield of dry and fresh matter of *Sesbania* plants after irrigation with BT water (Figure 1), but an improvement with NF and RO waters. This is related to the difference in concentration of salts in the soil [73]. Indeed, up to 6.66 dS/m (4.3 g/L) of salt in the soil solution (NF), the growth of the aerial parts of *Sesbania* is not affected, which is also observed in wheat varieties [74].

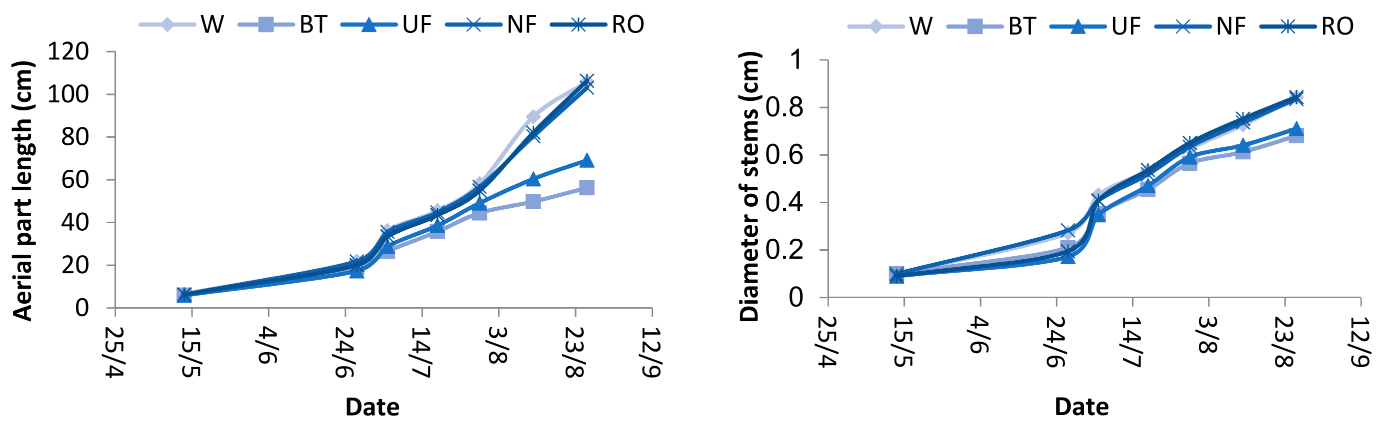


(**a**) (**b**)

Figure 1. Variation of fresh and dry weight of *Sesbania bispinosa* plants irrigated by TTE. (**a**) Variation of fresh weight of *Sesbania bispinosa* plants; and (**b**) variation of dry weight of *Sesbania bispinosa* plants. W: well water; BT: Biological Treatment; UF: Ultrafiltration; NF: Nanofiltration; and RO: Reverse Osmosis. The means of three observations followed by the different letters in each organ are significantly different according to Tukey's test at the level of $p < 0.05$.

3.3.2. Effect on the Aerial Parts

The growth in length and diameter of the plants' aerial parts is not influenced when irrigating with NF and RO waters (Figure 2). The variation of the stems' length and diameter appears to be gradual with time with an average length of the aerial part until the flowering stage of 106 cm, corresponding to normal growth. On the other hand, irrigation with BT and UF waters reduced plant growth.



(**a**) (**b**)

Figure 2. Growth evolution of the aerial parts of *Sesbania bispinosa*. (**a**) Aerial part length; and (**b**) diameter of stems. W: Well water; BT: Biological treatment; UF: Ultrafiltration; NF: Nanofiltration; and RO: Reverse Osmosis.

The impact of BT and RO water irrigation can be clearly distinguished by observing the plants at the end of the irrigation cycle (Figure 3). This impact is possibly due to soil salinity. Indeed, beyond 6.66 dS/m, which corresponds to the EC of the soil irrigated by NF waters, the growth of Sesbania plants decreased significantly. This effect was also found by Cherifi et al. [74].

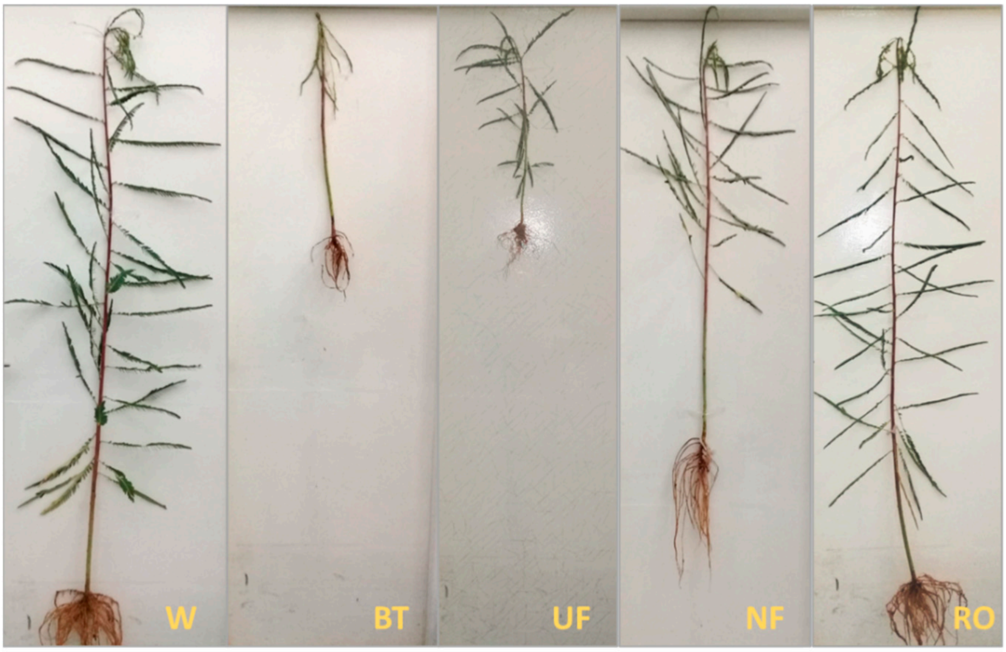


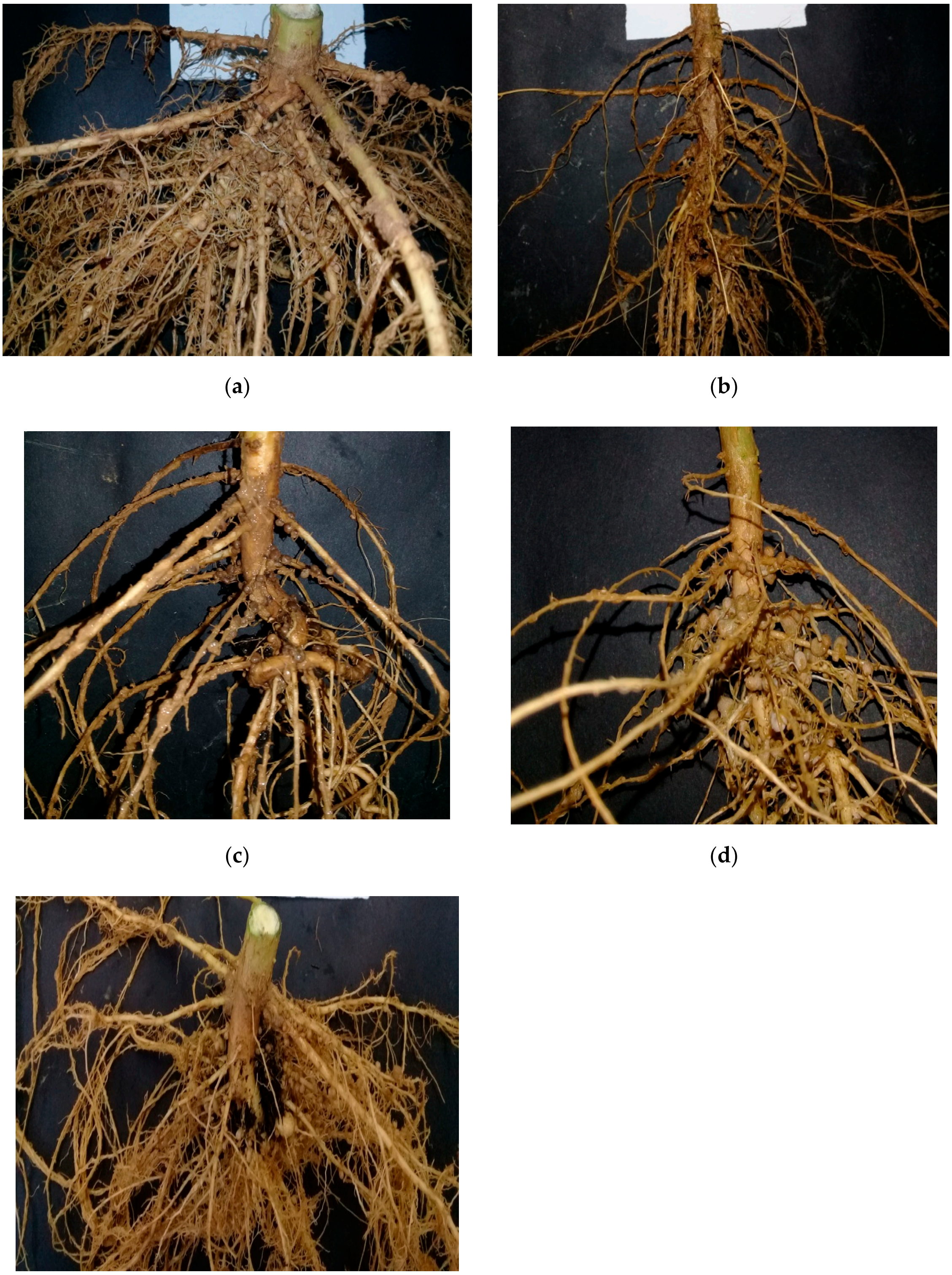
Figure 3. Effect of TTE irrigation on the growth of aerial parts of *Sesbania bispinosa*. W: Well water; BT: Biological treatment; UF: Ultrafiltration; NF: Nanofiltration; and RO: Reverse Osmosis.

3.3.3. Effect on the Root

The high frequency of nodule formation in the roots of *Sesbania* plants irrigated with well water (Figure 4a) is reduced with UF and NF waters (Figure 4c,d). Nodulation frequency was very low when BT water was used (Figure 4b). Thus, nodulation is sensitive to the different qualities of TTE, especially the high salinity of waters and soil. This effect on *S. bispinosa* is like that on alfalfa [75], *Sesbania sesban* [76], and durum wheat (*Triticum durum* Desf. "Massa") [77].

For plants irrigated with RO waters (Figure 4e), the granular sensation in the roots decreased slightly compared to control plants. In this case, the salinity problem no longer arises, and another factor is involved. This decrease can be attributed to the low concentration of sulfates in the soil and therefore low assimilation of this element by sesbania plants. Indeed, the assimilation of sulfates in plants is an essential pathway since they constitute a source of reduced sulfur for various cellular processes [78]. They play a key role in symbiotic nitrogen fixation (SNF) [79,80]. Sulfate deficiency has been shown to limit the development of root nodules due to low synthesis of nitrogenase, a bacterial nitrogen-reducing enzyme produced by nitrogen-fixing bacteroids [81–83]. This effect was observed by Varin et al. [79], who deduced that S deficiency in *Trifolium repens* reduced nodule development due to low nitrogenase production. Furthermore, Schneider et al. [80] suggest that sulfate deficiency has a direct impact on SNF and limits protein biosynthesis without adverse effects on the plant.

Thus, Allen and Allen [84] showed that the absence of nodules does not necessarily indicate the inability of the plant to enter an effective symbiosis. Several factors can negatively affect rhizobium populations and nodule formation, such as unfavorable soil type and soil pH, insufficient solar radiation, and extreme temperature conditions [84]. In the case of soybeans, Tu [85] showed that their inability to nodulate at high salt concentrations is due to a decrease in rhizobial colonization. Overall, root branching (root hairs) of *Sesbania* decreased with increased salinity in the soil solution compared to plants irrigated by well water.



(**a**) (**b**) (**c**) (**d**) (**e**)

Figure 4. Effect of TTE on the roots of *Sesbania bispinosa*. (**a**) Well water (W); (**b**) Biological Treatment (BT); (**c**) Ultrafiltration (UF); (**d**) Nanofiltration (NF); and (**e**): Reverse Osmosis (RO).

3.3.4. Effect on the Mineral Composition of Sesbania

The accumulation of salts in the soil is followed by the accumulation of Na^+ and Cl^- ions in *Sesbania* plants. Indeed, up to 6.66 dS/m (4.3 g/L), which corresponds to the ECe of soils irrigated by NF waters, *S. bispinosa* accumulated Na^+ in the aerial parts with low concentrations in the roots (Figure 5a). Beyond 8 dS/m of salt in the soil solution (BT and UF), *Sesbania* is under saline stress conditions and Na^+ concentrations increase significantly in roots and stems. The aerial parts exhibited the greatest quantity of Na^+, which may be a strategy of *Sesbania* to cope with salt stress [77]. A similar behavior was observed by Hajlaoui et al. [86] in two forage maize (*Zea mays* L.). The sequestration of Na^+ in the vacuole is an important and cost-effective strategy for an osmotic adjustment that reduces cytosolic Na^+ concentration [87,88]. It protects the cytoplasm, allows the plant to reduce its cellular water potential and, as such, prevents the loss of water [89]. 22

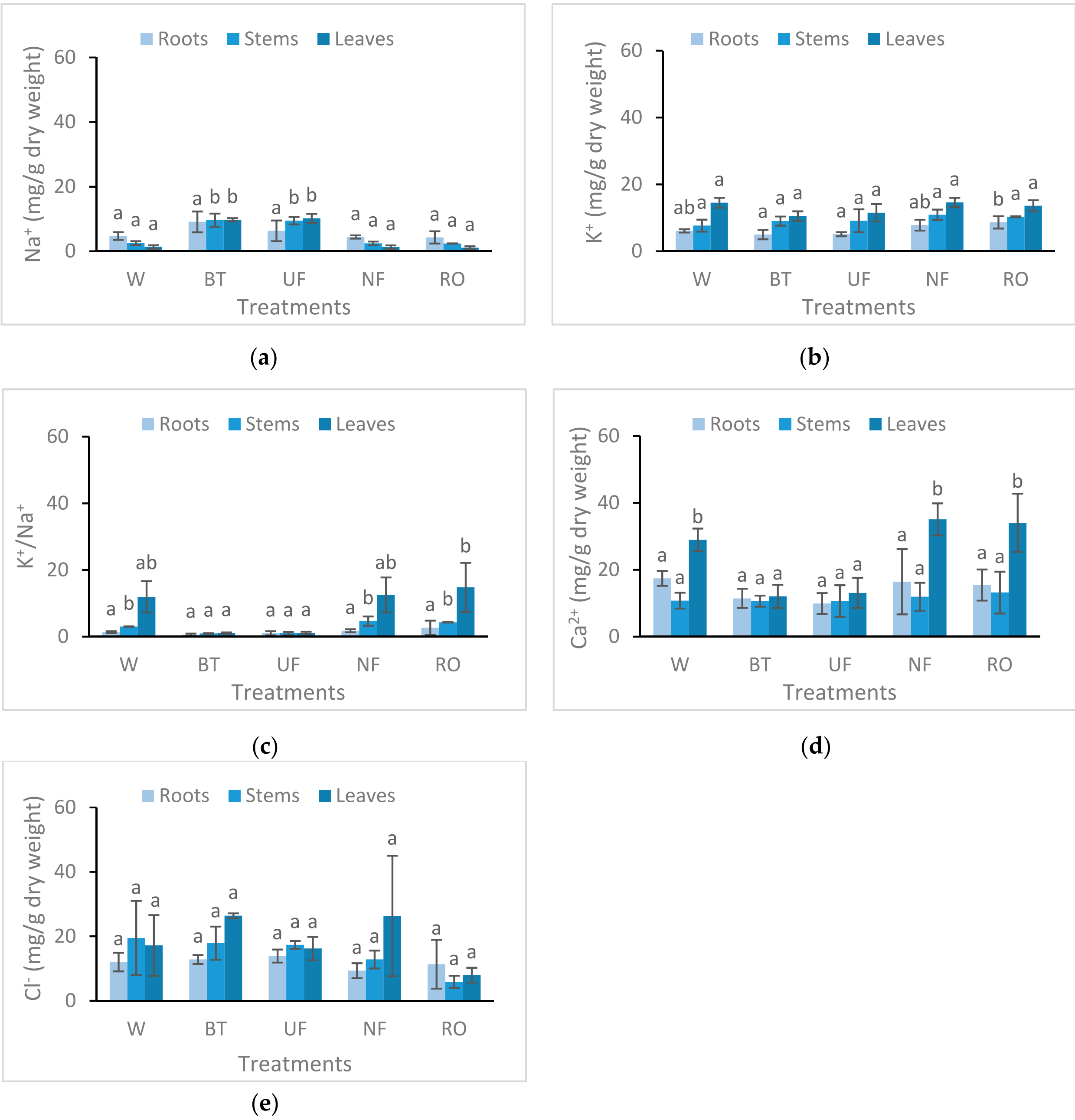


Figure 5. Effects of TTE on the mineral nutrition of Sesbania plants. (**a**) Na^+ concentration; (**b**) K^+ concentration; (**c**); K^+/Na^+ ratio; (**d**) Ca^{2+} concentration; and (**e**) Cl^- concentration. W: Well water; BT: Biological treatment; UF: Ultrafiltration; NF: Nanofiltration; and RO: Reverse Osmosis. The means of three observations (± standard deviation) followed by the different letters in each organ are significantly different according to Tukey's test at the level of $p < 0.05$.

In this present study, K^+ was mainly in the aerial part of *Sesbania* (Figure 5b) with unchanged concentrations compared to the reference plants but regardless of the concentrations in Na^+. The same effect was reported in the literature, where the absorption of K^+ decreases and can even stop with high doses of Na^+ [86,90,91]. Indeed, K^+ and Na^+ ions are always in competition to penetrate plant cells due to their physico-chemical similarity [92,93]. Thus, the K^+/Na^+ selectivity ratio is an index that describes the threshold of toxicity by Na^+, a high K^+/Na^+ ratio indicating less Na^+ toxicity [94,95].

Therefore, we established the K^+/Na^+ selectivity ratio. Our results show that the K^+/Na^+ ratio (Figure 5c) decreases with the increase in the salinity of the treated effluents and that of the soil. This ratio is lower in the roots than in the leaves. This is consistent with a previous study on tomatoes [96]. Cramer et al. [97] demonstrated that at high salt concentrations, Na^+ displaces Ca^{2+} from the plasmalemma of root cells, leading to an increase in membrane permeability, causing an efflux of K^+ and modifying the K^+/Na^+ selectivity ratio.

The highest levels of Ca^{2+} ions were recorded in the leaves of the plants, regardless of the irrigation water used (Figure 5d). A significant decrease in Ca^{2+} ($p < 0.05$) was observed in plants irrigated with BT and UF waters, whereas no negative effect was observed with NF and RO waters.

Cl^- ions content (Figure 5e) remained like those of the control plant, whereas it was reduced in the case of irrigation with RO waters and slightly increased in the leaves in the case of BT irrigation waters.

MTEs contents were under the detection limit and thus were not recorded neither in the aerial parts of *Sesbania*, nor in the roots, indicating that there was no translocation from soil to plant regardless of the irrigation water. This could be due to the sandy-loamy soil texture that reduces the available fraction of MTEs [98]. In relation to the MTE contents recorded in the soil, these results indicate that the MTEs are not assimilated by the plant, but rather leached by the drainage water.

4. Conclusions

Our work has made it possible to define the risks of textile effluents, the limit of the biological pre-treatment in progress in the factory, the constraints of the advanced treatment of membrane techniques, and to study the possibility of agricultural valorization of textile effluents treated through the choice of a fodder plant that can adapt to the quality of these waters. The results of this work studying membrane treatment processes for the decontamination of textile effluents and safe reuse in agricultural sector suggest that biological treatment alone cannot be considered neither to decontaminate textile effluents nor for reuse in irrigation, which requires additional treatments to improve their qualities. Our results find that additional treatments of these waters by Nanofiltration and Reverse Osmosis improve the physico-chemical quality of textile effluents and make them suitable for reuse in agriculture. Indeed, these two qualities of treated waters are coherent with the Tunisian standard NT 106.03 and can be reused in agriculture. Thus, according to the WHO guidelines on MTE levels in soil, no degree of restriction is required when irrigating with these waters. However, given the dominance of sodium and chloride ions in the soil, control measures must be taken to avoid the risk of soil salinization. We also deduce that the presence of MTEs in the waters of nanofiltration and reverse osmosis and in the soil did not affect the growth and mineral composition of *Sesbania*. As an alternative, given that the major problem of BT textile effluents is salinity and due to the high cost of RO treatment, softening by coupling with other water qualities such as well water is a simple and inexpensive solution to consider for sustainable unconventional water recovery. Couplings of membrane treatment processes can be envisaged. Thus, internal recycling is also a method to be developed by industrials. Finally, the choice of treatment of textile effluents and their reuse in agriculture, the sector that consumes the most water, appears beneficial for water-scarce countries because it reduces pressure on conventional water resources and limits discharges into the environment. On the other hand, the production of fodder irrigated by treated textile effluents by membrane processes can be envisaged in order to reduce the fodder deficit of livestock.

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Data Availability Statement: The data presented in this study are available on request from the corresponding author. The data are not publicly available because they are part of a thesis not yet defended.

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