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RESEARCH ARTICLE

# Demonstration scale chemical–physical treatment and agricultural reuse of highly saline textile wastewater

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## Abstract

This study aimed to develop an energy-efficient process for treating highly saline textile wastewater (TWW) in a 10 m$^3$/day pilot plant and evaluate forage sorghum irrigation with treated wastewater in terms of crop production and soil and irrigation device performance. The TWW treatment pilot plant, consisting of a coagulation/flocculation unit followed by a sand filter and an anion exchange resin column, produced treated effluent that complied with the permissible limits specified in the ISO 16075-2:2020 standard for Category C irrigation water. The corresponding average energy consumption was 1.77 kWh/m$^3$. Reusing treated TWW for forage sorghum irrigation over a 13-week cycle yielded crop performances comparable with freshwater irrigation, with no negative impact on the irrigation system. Although soil profiles were similar between treated TWW and freshwater irrigation, both soils featured an increase in electrical conductivity, which may reversibly or irreversibly affect soil quality and damage salt-sensitive crops. These findings demonstrate the effective treatment and reuse of saline TWW for irrigating salt-tolerant crops, offering significant implications for industrial wastewater management and cropping patterns in arid and semi-arid regions.

KEYWORDS
forage sorghum, irrigation, saline textile wastewater, soil, wastewater treatment and reuse, water management

## Highlights

- A 10-m$^3$/day pilot plant was developed for the treatment of highly saline textile wastewater.
- The pilot plant demonstrated average removal efficiencies of 63% for COD, 97% for colour, 96% for TSS and 21% for EC.

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**Abbreviations:** Cart, total carotenoids; Ch a, chlorophyll a; Ch b, chlorophyll b; COD, chemical oxygen demand; EC, electrical conductivity; EU, emission uniformity; FDA, fluoresceine diacetate hydrolysis activity; FW, freshwater; OM, organic matter; TDS, total dissolved solids; TSS, total suspended solids; TTWW, treated textile wastewater; TWW, textile wastewater; WW, wastewater; WWTP, wastewater treatment plants.

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- Treated effluent met ISO 16075-2:2020 standards for Category C irrigation water, with an average energy consumption of 1.77 kWh/m$^3$.
- The use of treated wastewater showed sorghum crop production comparable with freshwater irrigation.
- The use of treated wastewater had no adverse effects on the irrigation system; however, it led to an increase in soil electrical conductivity.

## 1 | INTRODUCTION

In recent years, population growth, rapid urbanization and climate change put enormous pressure on finite freshwater supplies, especially in arid and semiarid regions (Flörke et al., 2018; Leal Filho et al., 2022). Specifically, agriculture may be seriously affected if an alternative source of water is not available (Frascari et al., 2018). According to Ventura et al. (2019), about 70% of all the world's freshwater withdrawals are used for irrigation. The use of alternative water resources such as desalinated seawater and treated wastewater (WW) represents an effective water management strategy to complement conventional freshwater sources and sustain agricultural production (Suri et al., 2019).

In arid and semi-arid regions, water reuse is being increasingly practiced, particularly for irrigation, to address water scarcity challenges. Regulations for treated wastewater reuse vary depending on the specific region, country or jurisdiction. However, they generally involve stringent guidelines and standards to ensure that reclaimed water meets certain quality criteria before it can be reused. These regulations often dictate the permissible levels of various contaminants, such as suspended solids, nutrients, heavy metals and pathogens, in the treated wastewater. Additionally, regulations may outline requirements for monitoring, testing and reporting to ensure compliance with the established standards. Regulations may also address the distribution, storage and application of reclaimed water to minimize potential risks to human health and the environment (Mizyed, 2013).

In Tunisia, the reuse of treated WW has been adopted since the 1960s in many irrigated perimeters (Kefi et al., 2023). Tunisia has 125 WW treatment plants (WWTP) treating around 289 million m$^3$/year of WW. Around 22% of total treated WW are reused to irrigate 2734 exploited hectares, out of 8435 managed hectares. Treated WW is distributed to farmland, golf courses and green spaces, and it is also used for groundwater recharge (ONAS, 2022). Research conducted in Tunisia demonstrated relatively high levels of farmers' willingness to utilize reclaimed wastewater and the public's acceptance of consuming crops irrigated with such water (Akpan et al., 2020).

The textile industry is one of the largest sources of industrial WW, since it requires the use of high volumes of water in dyeing, printing and finishing processes (Nigam et al., 1996). Other significant sources of industrial WW include the chemical industry, food and beverage processing, and paper and pulp manufacturing (Singh et al., 2023). When comparing water footprints, the textile industry typically has a higher water footprint per unit of output compared with these other industries (Zhu et al., 2022). According to the Agency for the Promotion of Industry and Innovation (API) in Tunisia, around $2.8 \cdot 10^6$ m$^3$/year of textile wastewater (TWW) are generated by the Tunisian textile sector, accounting for 7% of the total industrial WW. Textile effluents usually have strong colour and contain a wide variety of organic and inorganic compounds (Azanaw et al., 2022; Methneni et al., 2021). During the dyeing processes, a portion of the applied dyes remains unfixed to the fabrics and gets washed out. To partially solve this problem, NaCl is used in the dyeing process to neutralize the fabrics' surface charge and improve dye adsorption.

In Tunisia, the quality standards for discharging TWW into the wastewater network are established by Tunisian decree n° 2018-315 (Table S1). If effluents fail to meet these criteria, they are required to undergo pre-treatment before entering the wastewater network. In cases where an emitter is situated outside of the intervention zone of ONAS (National Sanitation Utility of Tunisia) or if ONAS declines to accept its effluents, the emitter has the option to discharge its effluents directly into the environment (maritime public domain or hydraulic public domain), provided they comply with Tunisian decree (n° 2018-315) and obtain authorization from the relevant ministry overseeing their activity.

Several treatment technologies have been developed for highly saline TWW treatment, but the challenge remains to achieve high treatment performance with reduced energy consumption. Biological treatment methods offer cost-effective, non-toxic and sustainable solutions for treating TWW, effectively removing contaminants and reactive dyes. However, high salinity levels can induce saline shock, decreasing COD removal due to bacterial cell plasmolysis, and salt concentrations above 5000 mg/L may block the biological process by damaging microorganisms' cell walls (Guo et al., 2023). On the other hand, physico-chemical methods offer the potential for efficient treatment of saline wastewater by effectively removing organic matter and separating salt from water (Srivastava et al., 2021). Chemical oxidation processes like ozone ($O_3$) or hydrogen peroxide ($H_2O_2$) are commonly utilized for treating this type of wastewater. Despite their effectiveness in removing contaminants, they may not represent the most energy-efficient option, especially when considering the overall energy consumption and operational costs involved

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in the treatment process (Zhang et al., 2021). The coagulation-flocculation process is widely recognized as the most commonly utilized and highly effective method for treating highly saline TWW. Its advantage lies in its low energy consumption, making it an appealing choice for textile industries (Cui et al., 2020). However, one significant limitation of this method is its potential ineffectiveness in removing certain pollutants present in TWW, such as stable azo dyes, which may require additional treatment steps for complete removal (Zaharia et al., 2024). Other physical methods like adsorption and filtration might demonstrate limited dye removal efficiency in highly saline wastewater.

In general, there is no single treatment method that offers a universally effective solution for addressing TWW in terms of performance, energy efficiency and environmental sustainability. Hybrid processes integrating various treatment methods demonstrate considerable promise in enhancing overall treatment efficiency for highly saline TWW (Bener et al., 2020). Although there have been advancements in lab-scale studies for highly saline TWW treatment, large-scale implementations remain limited (Srivastava et al., 2021). In the present study, a continuous hybrid treatment system based on coagulation/flocculation, sand filtration and anion exchange resin processes has been developed and successfully demonstrated at a pilot scale for the treatment of highly saline TWW.

Although research emphasizes the importance of water reuse within the textile industry, the specific aspect of reusing highly saline-treated TWW (TTWW) in irrigation appears to be a poorly explored area (Pinto et al., 2022; Yin et al., 2019). Developing and validating appropriate treatments for the potential agricultural reuse of highly saline wastewater is thus a priority (Sahu, 2021). Simultaneously, the selection of salt-tolerant and low-water-intensive crops is an efficient strategy to enhance the agricultural reuse of TTWW. Forage sorghum, for example, is a hardy crop that can withstand a wide range of environmental stresses such as drought and salinity (Rooney, 2014). It is optimal for the feed of most livestock animals, since it provides a nutritional composition similar to that of corn, with a slightly higher energy content (3831 kcal/kg for sorghum against 3724 kcal/kg for corn) (Etuk et al., 2012). Studies on the effects of irrigation with saline industrial WW on forage sorghum plants performance and quality are extremely limited, especially in arid regions (Chaganti et al., 2020; Mabasa et al., 2021; Soni et al., 2016).

The present study aimed at (i) developing and assessing an integrated pilot-scale process for highly saline TWW treatment, (ii) estimating the total energy consumption of the developed treatment system and (iii) assessing the results of a field trial of forage sorghum irrigation with TTWW provided through micro-sprinklers, in terms of crop production performances, changes in soil quality and irrigation device performances.

The proposed integrated approach for highly saline TWW treatment and agricultural reuse could be implemented at a large scale in North Africa, leading to a marked decrease of the pollutant load discharged by textile industries in municipal treatment plants and to an increased availability of freshwater for agricultural and urban uses.

## 2 | MATERIALS AND METHODS

### 2.1 | Study area and textile wastewater characteristics

This study was conducted in the G-Wash textile industry located in Korba, within the Nabeul Governorate region, Tunisia. G-Wash produces 200 $m^3$/day of polluted TWW from dyeing and finishing activities.

The TWW generated by G-wash industry is characterized by its blue colour (5.4 ± 1.7 $m^{-1}$), which is attributed to a mixture of anionic dyes commonly used within this industry. These include Bezaktiv-S2G-Blue ($\lambda max = 600$ nm), Bezaktiv Blue S Matrix150 ($\lambda max = 522$ nm), Tubantin Brown GGL ($\lambda max = 416$ nm), Tubantin Blue BRR-HC ($\lambda max = 581$ nm), Bezaktiv Red S-Matrix ($\lambda max = 626$ nm) and Tubantin Orange GGLN 200 ($\lambda max = 410$ nm). Additionally, this wastewater is characterized by a high organic load (COD = 468 ± 119 mg/L), high concentrations of total suspended solids (580 ± 159 mg/L), low nutrient values ($NH_4$-N = 0.3 ± 0.2 mg/L, $PO_4$-P = 0.7 ± 0.3 mg/L) and high salinity (EC = 19.8 ± 4 mS/cm) owing to the use of saline groundwater and salts (mainly sulphate salts, up to 771 mg/L) (Table S1). Since the simultaneous effect of high salinity, recalcitrant dyes and lack of macro-nutrients plays an inhibitory role on biomass growth (Lellis et al., 2019; Uygur & Kargi, 2004), for this TWW, a chemical–physical treatment was considered more suitable than a biological treatment. All heavy metals were detected at trace levels and were largely inferior to the limits set by both the Tunisian standard for treated WW reuse in irrigation (NT106.03) and the Tunisian standard for TWW discharge into the public domain, as regulated by NT 106.02 (1989) and amended by Decree n° 2018-315 on 26 March 2018. No *E. coli* was detected in the TWW, confirming that the TWW generated by G-wash industry is not mixed with sanitary water.

### 2.2 | Treatment plant configuration and operation

The 10-$m^3$/day TWW treatment plant implemented in the textile dyeing industry G-wash included the following units (Figure 1): a 10-L coagulation/flocculation pipe, a 1.9-$m^3$ clarifier tank with a surface area of 1.12 $m^2$, a 0.6-$m^3$ sand filter, a 0.6-$m^3$ resin polishing filter connected to a brine tank containing the resin washing solution and a separate tank to collect the treated TWW for irrigation.

The optimization of the coagulant and flocculant dosage was carried out first using a laboratory bench-scale jar test. The experiments demonstrated that the optimal performance in terms of turbidity, COD and colour removal was achieved with the addition of 100 mg/L of coagulant (aluminium sulphate) and 5 mg/L of flocculant (CHTT Floc N, a chitosan-based flocculant).

The resin utilized in this research was the quaternized polyacrylic microspheres of NDMP, provided by Nanjing University, China. The NDMP is an anion exchange resin composed of 43.8% C, 13.2% N,

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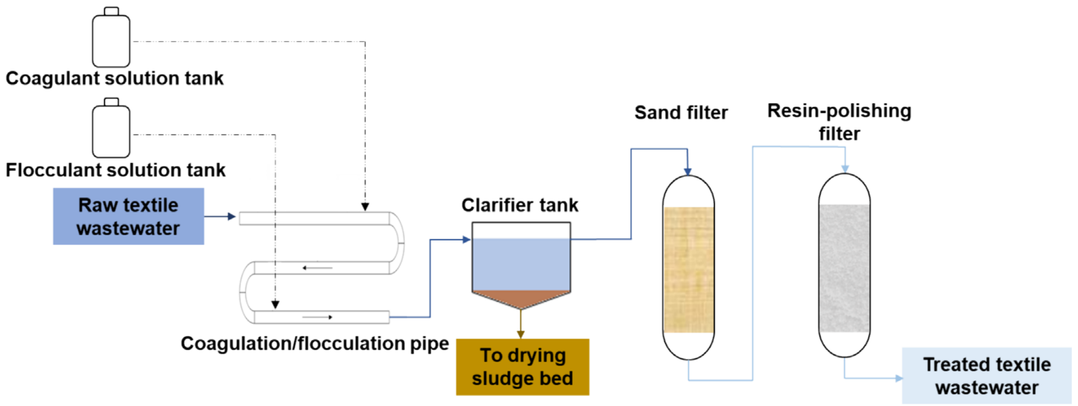

**FIGURE 1** Schematic diagram of textile wastewater treatment pilot plant.

24.2% O, 10.3% Cl and 7.6% Fe, as reported by Shuang et al. (2012). The selection of NDMP resin was based on its compatibility with the anionic nature of dyes commonly employed in the G-Wash industry, alongside its exceptional adsorption properties. Shuang et al. (2012) demonstrated that using the NDMP resin for the removal of reactive dyes, that is, Orange G and red RWO, resulted in higher adsorption capacity, faster kinetics and more effective regeneration when compared with the conventional quaternized resin MIEX. Additionally, Fan et al. (2014) reported that the NDMP resin exhibited higher efficiency in removing dissolved organic matter from real dyeing bio-treatment effluents compared with particle-activated carbon. Shi et al. (2015) also demonstrated NDMP ability to significantly reduce pollutant levels and mitigate toxicity in drinking water. The regeneration of the resin was carried out with a saturated NaCl solution, which was periodically renewed.

During the experimental period, the pilot plant was operated continuously at a flow rate of 10 $m^3$/day for 80 days, under the specified operating conditions: hydraulic retention time (HRT) coagulation-flocculation = 1.44 min, HRT clarifier tank = 4.56 h (surface hydraulic loading = 8.9 $m^3/m^2$/day), HRT sand filter = 2.4 h and HRT NDMP resin polishing filter = 2.4 h. The specified operating conditions were established through preliminary optimization of the HRT for each pilot plant unit, conducted before the start of the experimental period.

## 2.3 | Irrigation experimental design and treatments

Although several African countries face critical water shortages, many lack regulations and thus water reuse occurs without control measures. However, in Tunisia, the management of water reuse is guided by the Water Code of 1975, as well as subsequent decrees and standards introduced in 1989. Specific crops permitted for reuse and the corresponding project requirements were outlined in 1994 and 1995, respectively. Briefly, it is not allowed to irrigate vegetables consumed raw (with the exception of trees, forages, industrial crops and landscaping), and all reuse projects require a permit from the Ministry of Agriculture, the Ministry of Environment and Land Use Planning and the Ministry of Health. In the present study, treated TWW was used for the irrigation of a non-food crop, that is, forage sorghum.

For this research, forage sorghum [*Sorghum bicolor* (L.) Moench], a typical non-food crop largely grown in Tunisia, was selected for its high salinity tolerance compared with other forage species and its relatively short maturity period, which qualifies it as suitable for research purposes on treated WW reusability. In Tunisia, forage sorghum is typically planted once a year, usually during the spring or early summer months.

In the present work, irrigation experiments were conducted over a single 13-week cycle in a field near the G-wash industry. The field's soil is a loamy sand with the following composition, that is, sand 77.6%, clay 15.9% and silt 6.5%.

The irrigation experimental design included two water qualities, that is, TTWW and freshwater (FW). Both were provided through micro-sprinklers with the following features, that is, operating pressure of 1.0 bar, flow rate of 19 L/h and average drop size of 0.8 mm. The experiment was conducted in four replications, with a total of 2 treatments × 4 repetitions = 8 experimental plots of 3 m × 1 m = 3 $m^2$ size. The plots irrigated with TTWW were separated from each other by 1 m, whereas the plots irrigated with FW were separated from those irrigated with TTWW by 3 m in order to avoid any mixing between the two types of water. A total of 16 micro-sprinklers were used at the rate of two/plot (Figure 2). Sorghum seeds were sown at the rate of 378 seeds per plot. Each plot consisted of nine rows at 35 cm spacing with seed pockets (six grains per pocket) spaced 15 cm apart within rows. The Rain Bird ESP-RZXe programmer was used for irrigation scheduling.

During the first 3 weeks after sowing, irrigation was performed with low-salinity tap water in all plots, three times per week for 30 min at 1 bar, in order to avoid germination inhibition owing to the high salinity of TTWW (Mbinda & Kimtai, 2019). During the rest of the growth cycle (10 weeks), the plants were irrigated at the same rate in accordance with the layout described above using TTWW and FW as control. The FW used during the experimental period came from groundwater sources and was characterized by a neutral pH (7.64), a high salinity (EC = 17.28 mS/cm, total dissolved solids

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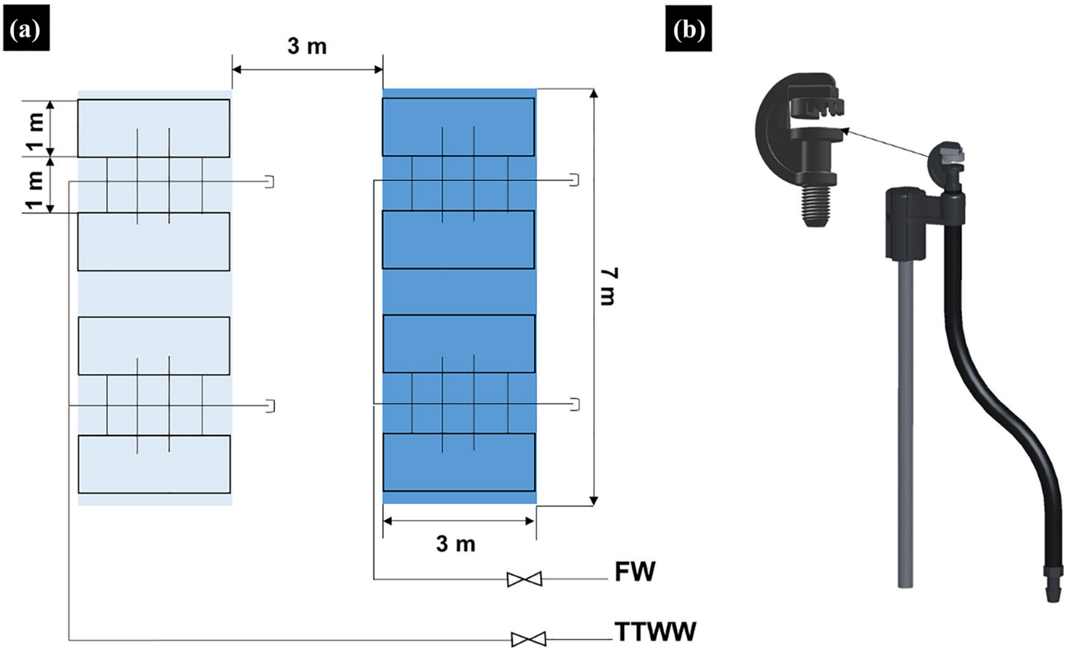

**FIGURE 2** (a) Schematic diagram for the irrigation network layout; (b) microsprinklers. FW, freshwater; TTWW, treated textile wastewater.

[TDS] = 12.81 g/L), a nitrate concentration of 3.5 mg/L and low orthophosphate and ammonia levels (<0.05 mg/L). The high salinity levels in the groundwater of this region are primarily attributed to seawater intrusion resulting from the overexploitation of aquifers (Ayari et al., 2023).

The irrigation system was stopped each time the rainfall was sufficient to cover the water need of the crops. Irrigation was avoided during windy conditions to maintain irrigation distribution uniformity. Taking into account the initial 3 week of irrigation with tap water, the total amounts of water supplied were 555 $m^3$/ha of tap water + 1665 $m^3$/ha of TTWW in the TTWW plots, and 555 $m^3$/ha of tap water + 1665 $m^3$/ha of FW in the FW control plots.

## 2.4 | Analytical methods

### 2.4.1 | Wastewater sampling and analyses

Samples were periodically collected from the inlet (equalization basin) and outlet of the pilot plant in clean plastic containers and transferred immediately to the laboratory for characterization. Chemical oxygen demand (COD), $NH_4$-N and $PO_4$-P concentrations were assessed using Hach Lange test cells (LCK 400, 304 and 349, respectively) via a spectrophotometer (DR 1900, Hach Lange), following ISO standards 15705:2002, 7150-1:1984 and 6878:2004, respectively. The samples were filtered through qualitative filter paper prior to analysis. Electrical conductivity (EC) and pH levels were assessed using the Loviband SensoDirect 150 multi-parameter water quality meter. Biochemical Oxygen Demand ($BOD_5$) and total suspended solids (TSS) were analysed following standard methods for the Examination of Water and WW (APHA, 2005). The area of the absorbance spectrum in the 400- to 800-nm region was registered for each sample to determine the decolorization efficiency of the pilot plant. Faecal and total coliforms were measured using the multiple-tube fermentation method (APHA, 2005). These standardized methods ensured accurate and reliable data for the analysis of the inlet and outlet samples taken during the monitoring process.

### 2.4.2 | Soil sampling and analyses

Soil samples were taken before sowing and at the end of the experimental period. Composite soil samples from 0- to 20-cm depth were prepared by air drying at 25–30°C and sifted through a 2-mm mesh sieve. The samples were analysed in terms of pH, EC, organic matter (OM) and microbial activity (fluoresceine diacetate hydrolysis [FDA] activity). The soil pH (1:2.5 w/v soil:water suspension) and EC (1:5 w/v soil:water suspension) were measured using a type Loviband SensoDirect 150 multiparameter probe. Soil OM was measured using the weight loss on ignition method (Cambardella et al., 2001). FDA was determined by measuring the fluorescein released after hydrolysis of fluorescein diacetate following the method outlined by Prosser et al. (2011).

### 2.4.3 | Crops sampling and analyses

At 13 weeks after sowing (grain maturity stage), sorghum plants were sampled randomly and studied. Shoot length, number of leaves, diameter of the main stem and fresh and dry weight of shoots were

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measured. Plant length was determined by measuring the length of the plant from ground level to the tip of top leaf. Sorghum plants were cut at the ground to constitute fresh biomass. To determine dry biomass, fresh biomass was steamed at 70°C for 72 h and weighed. The contents of chlorophyll a (Ch a), chlorophyll b (Ch b) and total carotenoids (cart) were determined in aqueous acetone (80%) extracts of green plant tissue by UV–VIS spectroscopy (Arnon, 1949).

### 2.4.4 | Irrigation system distribution performance

To evaluate emitter performance, the flow rate was determined for each emitter by measuring the volume of effluent, using a 100-mL graduated cylinder, over a fixed unit of time.

Emission uniformity (EU) is a measure of how uniformly water is applied to the irrigated area. It is measured by averaging the lowest quarter (25%) of the discharge samples to the global average (Equation 1):

$$\mathrm{EU}(\%) = 100 \cdot \left(\frac{q'_n}{q'_a}\right) \tag{1}$$

where

- $q'_n$: average discharge flow rate of the lowest 25% of the field data discharge readings (L/h)
- $q'_a$: average discharge flow rate of all the field data emitter discharges (L/h)

## 2.5 | Energy consumption of the TWW treatment pilot plant

Energy consumption refers to the relative amount of energy needed per unit volume of wastewater for tasks related to water management, including water treatment and pumping (Copeland, 2014; Navigant Consulting Inc., 2006). In this study, energy consumption was calculated based on the energy demands associated with the operation of pumps and solenoid valves.

The electrical energy input was estimated by considering the electrical power consumption (kW) of the pumps (i.e., influent feed pumps, coagulant and flocculant dosing pumps, sludge pump and filter feed pumps) and solenoid valves (one for resin regeneration and backwash, and another for sand backwash), the time in hours (h) for which the pumps and solenoid valves were operated and the total amount of wastewater treated (Equation 2).

$$Ep = \frac{P \times T}{Q} \tag{2}$$

where $E_p$ is the electrical energy kWh/m$^3$, $Q$ is the total flow of wastewater in m$^3$/day, $P$ is the rated power of the electrical pumps and solenoid valves in kilowatt (kW) and $T$ is the operation hours in a day (h/day). Table 1 presents the specifications of the pumps and solenoid valves.

## 2.6 | Statistical analysis of data

Experimental data were statistically analysed using the one-way analysis of variance (ANOVA) followed by a Bonferroni post hoc test using the IBM SPSS Statistics 21 software package.

# 3 | RESULTS AND DISCUSSION

## 3.1 | Textile wastewater treatment effectiveness

The average removal efficiencies and effluent characteristics relative to the TWW treatment process are reported in Table 2 along with the relevant standards for WW discharge and reuse. These standards encompass the Tunisian National standards for TWW discharge into the public domain (NT 106.02, 1989, as amended by Decree n° 2018-315) and for the reuse of treated WW for irrigation (NT 106.03), as well as the ISO 16075-2:2020 standard on which the EU 2020/741 regulation for treated WW reuse in agriculture was based.

TSS and COD are two important performance parameters for determining the suitability of treated wastewater for irrigation. A high level of biodegradable COD may encourage microbial development within the system, which may lead to emitter clogging in the irrigation network. Similar to this, high levels of suspended solids would cause severe damage to the irrigation system, especially if drip irrigation was used. The irrigation network may get clogged as a result of the accumulation of suspended solids as deposits in the irrigation tubing and water emitters. The presence of suspended solids in TWW is mainly attributed to fibres, lint, dye particles and other solid materials (Christian et al., 2023). Decantation alone is often inefficient for removing fibres because of their colloidal nature, negatively charged surfaces and slow settling rates. In this context, coagulation-flocculation emerges as a favourable solution for addressing the removal of fibres and suspended solids in TWW (Azanaw et al., 2022).

Based on the composition of the inlet and outlet, a COD mass balance was performed to analyse the system performance. It was

**TABLE 1** Details of pumps and solenoid valve specifications.

| Treatment unit | No. of working units | *P* (kW) | *T* (h/day) |
|---|---|---|---|
| Feed pump | 2 | 0.75 | 8 |
| Dosing pump | 2 | 0.19 | 3.2 |
| Sludge pump | 1 | 0.75 | 0.2 |
| Filter feed pump | 2 | 0.38 | 5.6 |
| Solenoid valve | 2 | 0.05 | 0.3 |

*Note*: *P*, rated power of the electrical pump in kilo Watt; *T*, operation hours in a day.

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**TABLE 2** Inlet/outlet concentrations and removal efficiencies of some parameters of samples collected before and after treatment, thresholds for the reuse of treated wastewater according to the Tunisian and the ISO 16075-2:2020 standards.

|  |  |  |  |  |  | ISO 16075 Cat. A[c] |  | ISO 16075 Cat. B[c] |  | ISO 16075 Cat. C[c] |  | ISO 16075 Cat. D[c] |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Parameter | Inlet | Outlet | Removal efficiency | Tunisian decree (n° 2018–315) for discharge[a] | NT 106.03 for reuse in irrigation[b] | Avg/95% ile[d] | Max | Avg/95% ile[d] | Max | Avg/95% ile[d] | Max | Avg/95% ile[d] | Max |
| pH | 7.4 | 6.7 | - | 6.5–9.0 | 6.5–8.5 | - | - | - | - | - | - | - | - |
| EC (mS/cm) | 19.8 | 15.6 | 21% | - | 7 | - | - | - | - | - | - | - | - |
| COD (mg $O_2$/L) | 468 | 176 | 63% | 160 | 90 | - | - | - | - | - | - | - | - |
| $BOD_5$ (mg $O_2$/L) | 89 | 31 | 65% | 30 | 30 | ≤5 | 10 | ≤10 | 20 | ≤20 | 35 | ≤60 | 100 |
| TSS (mg/L) | 580 | 25 | 96% | 50 | 30 | ≤5 | 10 | ≤10 | 25 | ≤30 | 50 | ≤90 | 140 |
| $NH_4$-N (mg/L) | 0.3 | 0 | 100% | 10 | - | - | - | - | - | - | - | - | - |
| $PO_4$-P (mg/L) | 0.7 | 0 | 100% | 2 | - | - | - | - | - | - | - | - | - |
| Colour (area of absorbance spectrum 400–800 nm) | 7.2 | 0.2 | 97% | - | - | - | - | - | - | - | - | - | - |
| Total coliforms (MPN/100 mL) | 240 | ND | >2.38 $\log_{10}$ units | - | - | - | - | - | - | - | - | - | - |
| Faecal coliforms (MPN/100 mL) | 7.5 | ND | >0.87 $\log_{10}$ units | 2000 | - | ≤10 | 100 | ≤200 | 1000 | ≤1000 | 10 000 | - | - |

*Note*: ND, not detected (<1 MPN/100 mL).

[a] Tunisian decree (n° 2018-315) for TWW discharge in the maritime public domain.

[b] NT 106.03: Tunisian Standard for irrigation with treated wastewater.

[c] ISO 16075-2:2020: Guidelines for treated wastewater use for irrigation projects. Cat. A: water suitable to irrigate food crops that are consumed raw. Cat. B: water suitable to irrigate food crops that are consumed after cooking. Cat. C: Agricultural irrigation of non-food crops. Cat. D: Restricted irrigation of industrial and seeded crops.

[d] Avg/95%ile: Avg for $BOD_5$ and TSS; 95%ile for faecal coliforms.

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found that 63% of the incoming COD was removed through accumulation in the sludge during the coagulation-flocculation step and via the adsorption process. Notably, the average outlet COD concentration of 176 mg/L slightly exceeds the limits set by Tunisian National standards.

The mean outlet TSS and $BOD_5$ concentrations (25 and 31 mg/L, respectively) were within the limits set by the Tunisian National Standards. The treatment led to a complete removal of orthophosphate and ammoniacal nitrogen and to a 97% colour removal (Figure 1S). The TTWW was found to be of high hygienic quality, thanks to removal of total and faecal coliforms to below the limit of detection (1 MPN/100 mL).

The observed pH values before and after treatment were within the permissible limits for water reuse in agriculture set by standard NT106.03 and within the Tunisian acceptable WW discharge range of 6.5–9.0. TWW treatment resulted in a minor but significant reduction ($p = 0.0004$) in EC values, from 19.8 mS/cm in raw TWW to 15.6 mS/cm after treatment. The EC decline can be mainly attributed to the efficient performance of both the coagulation-flocculation unit and the anion exchange resin. The coagulation-flocculation process contributes to eliminating dissolved organic and inorganic substances, including certain salt ions, which could be associated with suspended particles or colloids. As TWW passes through the anion exchange resin column, the anionic ions found in the wastewater, like sulphate, are drawn towards and captured by the positively charged functional groups of the resin.

Salt from highly saline TWW can be efficiently recovered and recycled through methods such as membrane separation processes like nanofiltration (NF) and selective crystallization. NF effectively separates salts from the wastewater, enabling their recovery and reuse in internal applications of textile industries. Meanwhile, selective crystallization is another method that can achieve high purity levels exceeding 90% by crystallizing salts from the wastewater. This approach is particularly useful for obtaining high-purity salts that can be reused in various industrial processes (Guo et al., 2023; Kieselbach et al., 2020).

In all, the treated TWW complied with the majority of the limits outlined in the Decree n° 2018-315 for TWW discharge into the public domain and the national standard for reusing treated wastewater in irrigation (NT 106-03), except for EC and, to a lesser extent, COD. The regulations outlined in Decree No. 2018-315 set the following limits: COD at 160 mg/L, $BOD_5$ at 30 mg/L, TSS at 50 mg/L and faecal coliforms at 2000 MPN/100 mL. Meanwhile, NT 106.03 specifies the following criteria: EC at 7 ms/Cm, COD at 90 mg/L, $BOD_5$ at 30 mg/L and TSS at 30 mg/L. Additionally, treated TWW complied with the ISO 16075-2:2020 standard for category C irrigation water, corresponding to the irrigation of non-food crops. This standard sets maximum concentrations for $BOD_5$, TSS and faecal coliforms at 35 mg/L, 50 mg/L and 10 000 MPN/100 mL, respectively. Although $BOD_5$ values remained below the maximum threshold of 35 mg/L, achieving an average $BOD_5$ reduction from 31 to 20 mg/L would be required.

Treated WW has been widely utilized for irrigation of non-food crops in various countries. For instance, Tunisia applied treated WW for irrigating fodder (alfalfa, sorghum and berseem), industrial crops (sugarbeet) and cereals, as discussed in research by Cherif et al. (2018). In Jordan, treated WW was utilized for irrigating non-food crops like fodder crops, that is, alfalfa (annual and perennial) and ryegrass, as highlighted by Abu-Awwad (2021). Egypt explored the use of treated WW for irrigating green areas of educational establishments and public and private parks (such as fence plants and flowers), as examined by Yehia and Mamdouh (2022). Mexico engaged in the large-scale use of treated WW for irrigating fodder crops, namely, fodder oats, fodder barley, grass, sorghum, alfalfa and forage corn, as researched by Valdes Ramos et al. (2019). United Arab Emirates utilized treated WW to grow fodder, fibre, pastures, seeds and some nursery crops, as discussed in studies by Al Hamedi et al. (2023).

## 3.2 | Energy efficiency of the TWW pilot plant

The treatment of TWW requires considerable energy consumption, primarily attributable to the need for intensive processes to remove contaminants and pollutants from the wastewater. These processes often involve advanced treatment technologies such as electrochemical catalysis, membrane bioreactors and other energy-intensive methods. The need for energy-efficient processes to achieve the desired levels of pollutant removal and water quality improvement in TWW treatment remains a significant challenge (Azanaw et al., 2022).

There is a lack of sufficient data in the literature for comparison, and the few available studies report values that vary significantly because of the difference in scale and scope of the study. In the present work, the energy needs of the developed system were estimated at 1.77 $kWh/m^3$ of treated water, achieving a COD removal rate of 63%. Although achieving relatively lower COD removal compared with other studied treatment processes, the implemented system nonetheless demonstrated significantly higher energy efficiency. For instance, in their study on real silk TWW treatment using 2-D electrochemical coagulation, Hemalatha and Shekar Sanjay (2023) reported an energy consumption of 2115 $kWh/m^3$ to achieve an 80% removal of COD. The 2-D electrochemical coagulation involves the application of an electrical potential to induce coagulation of suspended particles in water, typically using a two-dimensional electrode configuration. This process promotes the formation of metal hydroxide flocs, which aid in the removal of contaminants through aggregation and subsequent settling (Hemalatha & Shekar Sanjay, 2023). Aquino et al. (2014) found that the electrochemical coagulation of real TWW using $Ti–Pt/\beta–PbO_2$ electrodes required an electrical energy consumption of 50 $kWh/m^3$ to achieve nearly complete removal of COD. In a study carried out by Kuleyin et al. (2021), the advanced treatment of biologically treated TWW employing graphite electrodes exhibited an energy consumption of 16 $kWh/m^3$ to achieve 93% of COD removal. On the other hand, only one study, conducted by Dalvand et al. (2011), reported slightly lower energy consumption with electrocoagulation

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treatment for TWW, achieving an 84% COD removal with 1.30 kWh/m$^3$. The low energy consumption of the developed pilot plant compared with the processes mentioned earlier can be explained by the efficient use of energy solely for the operation of the pumps and solenoid valves.In comparison with the aforementioned methods, the developed TWW treatment system utilizes a range of chemicals, including coagulant and flocculant for the coagulation-flocculation unit and anionic resin along with NaCl for regeneration of the anion exchange resin column. Although these chemicals incur additional costs, they were selected for their cost-effectiveness and their ability to achieve the desired treatment objectives. Despite the slightly lower COD removal compared with some other treatment methods, the developed pilot plant demonstrated a balanced approach, prioritizing both economic and energy feasibility alongside the achievement of treatment objectives.

## 3.3 | Potential use of TTWW in irrigation

### 3.3.1 | Effect of irrigation with TTWW on morphological, physiological and agronomic parameters of sorghum plants

Few studies in the literature have systematically assessed the potential of TTWW for irrigation or the effects on soil chemical composition and plant growth (Bener et al., 2020; Dhaouefi et al., 2018; Okcu et al., 2019).

In our particular study, the effect of irrigation with TTWW on selected growth parameters, that is, shoot length, number of leaves, diameter of the main stem and fresh and dry weight of shoots of sorghum plants, was found to be statistically insignificant ($p > 0.05$), compared with FW (Figure 3).

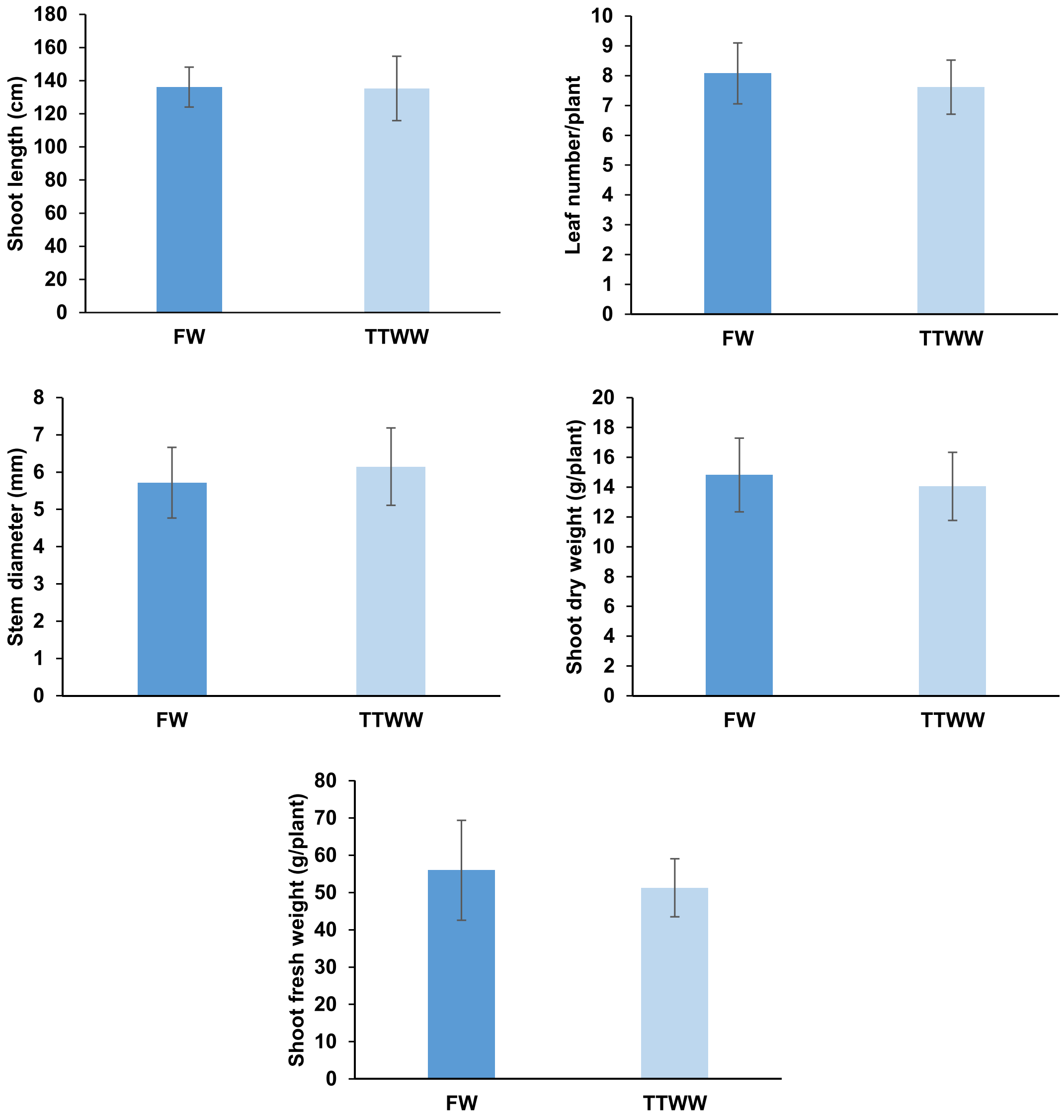

**FIGURE 3** Effect of fresh water (FW) and treated textile wastewater (TTWW) on shoot length, number of leaves, diameter of the main stem and fresh and dry weight of shoots of sorghum plants.

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The pigment contents in sorghum leaf also exhibited a similar trend. Figure 4 shows the results of analyses of chlorophyll a and b and carotenoid pigments extracted from sorghum leaves sampled from control and TTWW-irrigated plots. The differences between the FW and TTWW plots were not significant ($p > 0.05$) for all pigments. Carotenoid pigment production in forage sorghum primarily occurs through biosynthetic pathways within plant cells, namely, the MEP (methylerythritol phosphate) pathway and the Mevalonate pathway (Salas Fernandez et al., 2008). Commercially, carotenoids from forage sorghum find use in feed additives for livestock, nutritional supplements, pharmaceuticals, cosmetics and food colouring because of their health benefits, antioxidant properties and colour enhancement capabilities (Li et al., 2021).

Regarding the sorghum yield parameters, the results depicted in Table 3 indicate that there were no significant differences ($p > 0.05$) in sorghum biomass, grain yield, biomass and grain water productivity attributes between the FW and TTWW irrigated plots. These findings show that sorghum plants remained unaffected by TTWW irrigation. These results align with those reported by Bener et al. (2020), who employed TTWW for irrigating grass seeds and found a close similarity in the length of grass irrigated with both TTWW and tap water. Contrarily, Garg and Kaushik (2007) reported a decrease in growth parameters of two cultivars of sorghum when irrigated with untreated and TTWW at high TTWW:FW ratios (i.e., 50:50, 75:25 and 100:0). The effect was more pronounced for the untreated effluent. Likewise, another study examining the influence of wastewater irrigation on wheat yields found that untreated TWW negatively affected the growth and yield of wheat (Sahar et al., 2017). On the other hand, a study by Dhaouefi et al. (2018) demonstrated enhanced growth parameters in *Raphanus sativus* plants irrigated with both treated and untreated synthetic TWW compared with those irrigated with fresh water. In our study, in addition to the good quality of TTWW, both TTWW and FW display high levels of salinity, which may explain the similar behaviour of plants irrigated with FW and TTWW. Sorghum is well known for its strong resistance to abiotic stress and wide adaptability. Salt tolerance is one of its main characteristics. It is able to successfully cope with osmotic stress caused by the excess of NaCl and to tolerate salinity levels of up to 15 mS/cm during late growth stages (Hussien Ibrahim et al., 2020; Verheye, 2009). Notably, from a sanitation perspective, the excellent sanitary quality of the TTWW employed for irrigation, along with the absence of heavy metals, could potentially have a beneficial impact on the health of sorghum plants intended for consumption.

### 3.3.2 | Effect of TTWW on soil

Following the 13-week irrigation experiment, the effects of TTWW on the soil's physicochemical properties and microbial activity were assessed and are presented in Table 4.

The original soil is slightly alkaline with a pH of 7.9. After irrigation with both TTWW and FW, there was a negligible increase in soil pH, reaching 8.1 ($p > 0.05$). Soil pH affects the availability of nutrients in the soil. Between pH 6.0 and 7.0, most plant nutrients are in their most available state, whereas alkaline soils require special nutrient management techniques and the addition of acidic organic matter. Irrigation with TTWW and FW resulted in a marked increase in EC, from 0.49 to 1.48 mS/cm and 1.14 mS/cm, respectively. This increase, attributed to the high levels of TDS in TTWW and FW, may be critical for salt-sensitive crops and could potentially degrade soil quality either reversibly or irreversibly. The cation that most significantly influences salinity is sodium, especially when it is in excess of 100 mg/kg in the soil. It can be leached from the soil, thereby reducing the EC levels, through the addition of elemental sulphur or gypsum (Qadir et al., 2001; Thaker et al., 2021). If excessive salt is not adequately removed, it can accumulate in the soil, particularly in the topsoil due to high evaporation rates (Okcu et al., 2019). Improper use of

**TABLE 3** Effect of irrigation with FW and TTWW on yield and water productivity of sorghum.

| Agronomic parameters | FW | TTWW |
|---|---|---|
| Grain production yield (kg grain/ha) | 1487 ± 48 | 1404 ± 81 |
| Biomass production yield (kg biomass/ha) | 18 648 ± 102 | 17 701 ± 95 |
| Grain water productivity (kg grain/$m^3$ water supplied) | 0.67 ± 0.02 | 0.63 ± 0.01 |
| Biomass water productivity (kg biomass/$m^3$ water supplied) | 8.40 ± 0.65 | 7.97 ± 0.77 |

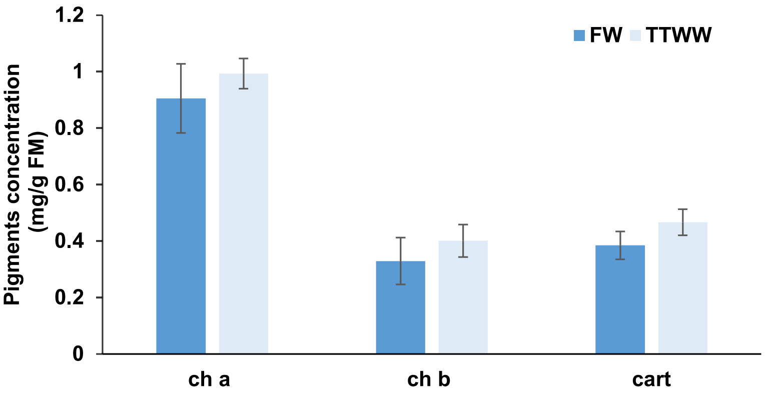

**FIGURE 4** Chlorophyll a (ch a), b (ch b) and carotenoid (cart) concentrations in leaves of sorghum plants irrigated with fresh water (FW) and treated textile wastewater (TTWW) FM fresh matter.

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**TABLE 4** Physicochemical properties and microbial activity of the soil before and after experiment.

| Parameters | Original soil | Soil irrigated with FW | Soil irrigated with TTWW |
|---|---|---|---|
| pH | 7.9 ± 0.09 | 8.1 ± 0.06 | 8.1 ± 0.05 |
| EC (mS/cm) | 0.49 ± 0.04 | 1.14 ± 0.02 | 1.48 ± 0.03 |
| OM (%) | 2.82 ± 0.12 | 3.17 ± 0.26 | 2.98 ± 0.18 |
| FDA (μg fluorescein/g dry sol/30 min) | 1.65 ± 0.12 | 2.35 ± 0.16 | 2.08 ± 0.19 |

brackish and saline irrigation water, inadequate drainage, rising water tables and other factors contribute to the salinization of land and water resources (Ma et al., 2023). The presence of excess salts can impact the metabolism of soil flora and fauna, ultimately resulting in the depletion of soil life and the transformation of once fertile and productive lands into barren and desert areas (Thaker et al., 2021). Our results are supported by Arif et al. (2016), who reported that irrigation with untreated TWW had no significant effect on pH values of the soil, whereas EC increased significantly.

No significant change was observed in soil organic matter (OM) after irrigation with TTWW or FW. However, a number of field studies reported an increase in soil OM in long-term WW-irrigated soils (Friedel et al., 2000; Zhang et al., 2023).

The FDA activity is widely accepted as an accurate and simple measurement of total microbial activity in soils and includes the ubiquitous free and membrane-bound digestion enzymes, such as lipase, protease and esterase enzymes. A slight increase in FDA activity ($p > 0.05$) was observed in both soils irrigated with FW and TTWW, compared with the soil before planting (Table 4), indicating that neither TTWW nor FW had detrimental effects on soil microbial activity. The increase of microbial activity in the soil around the roots indicates the strong influence of root hairs as potential drivers of microbial activity (Quattrone et al., 2024). Nonetheless, Pokhriya et al. (2020) reported adverse effects on soil ecology and health resulting from the use of textile effluent for irrigation. The presence of contaminants such as COD, BOD, pH fluctuations, dyes and heavy metals were identified as contributing factors.

These findings provide promising evidence for the possible use of TTWW in irrigation without any compromise on physiological and growth parameters of sorghum plantlets as well as on soil quality. However, the long-term effects of increased EC may impact salt-sensitive crops and degrade soil quality either reversibly or irreversibly.

### 3.3.3 | Effect of TTWW on the micro-sprinklers discharge

The effect of wastewater on micro-sprinklers' discharge has been studied in the context of micro-irrigation systems. Research has shown that the use of treated wastewater in micro-irrigation can lead to issues such as clogging of micro-sprinklers because of biofilm development and chemical precipitation (Rizk et al., 2021). Interestingly, in the present study, the average discharge flow rate values recorded for each emitter exhibited no significant difference ($p > 0.05$) in flow rate between the application of TTWW and FW (Table S2). At a pressure of 1 bar, the average discharge of the emitters was found to be 17.4 and 18.5 L/h in areas irrigated with FW and TTWW, respectively, very close to the manufacturing discharge (19 L/h).

Throughout the experimental period, the EU of the micro-sprinklers was monitored to ensure proper functioning and equal distribution of irrigation water to all plants. The estimated EUs under FW and TTWW irrigation were found to be 91% and 84%, respectively. An EU lower than 60% is considered too low, whereas it is generally recommended to exceed 75% (Li et al., 2024). All emitters were classified as 'excellent' based on the ASAE Standard EP405.1 (ASAE Standard, 2003), which recommends an EU between 80% and 90%. The slight EU diminution observed in areas irrigated with TTWW could be attributed to the clogging of some emitters owing to the high salt content. All emitters have to be cleaned up every one to 2 weeks in order to prevent their clogging and to maintain proper functioning.

## 4 | CONCLUSIONS

The findings of this study offer practical solutions for two critical challenges: TWW treatment and sustainable agricultural practices, particularly in arid regions facing water scarcity and saline wastewater disposal challenges.

Firstly, the development of an energy-efficient treatment process for highly saline TWW provides a viable option for industries burdened with managing saline effluents. The developed pilot plant, integrating coagulation-flocculation, sand filtration and anion exchange, effectively purifies wastewater, yielding a treated effluent meeting ISO 16075-2:2020 standards for Category C irrigation water. With an average energy consumption of 1.77 kWh/m$^3$, this process stands as an economically feasible solution for wastewater treatment, potentially reducing the ecological footprint of industrial operations. Notably, considering local electricity cost (1 kWh = 0.210 Tunisian Dinar = 0.067 US Dollar), the cost of 1 m$^3$ of treated water amounts to 0.118 US dollars. This cost is significantly lower than the price of 1 m$^3$ of tap water, which stands at 0.35 US dollars if the quarterly consumption falls within the range of 40 to 70 m$^3$. However, if consumption exceeds 150 m$^3$, the price can increase to 0.75 US dollars.

Secondly, the successful reuse of treated TWW for irrigating forage sorghum demonstrates the integration of wastewater management with agricultural practices. By substituting freshwater with TTWW, precious water resources are conserved, while sustaining crop production. The comparable performance of forage sorghum under TTWW and FW irrigation suggests that salt-tolerant crops can grow under these conditions, offering a resilient cropping alternative for regions constrained by freshwater availability.

Furthermore, the study underscores the necessity of monitoring soil quality and crop response to TTWW irrigation. Although no adverse effects on crop performance or irrigation system integrity were observed over a 13-week cycle, future research should prioritize long-term assessments of soil health. This includes evaluating the potential reversible or irreversible impacts of elevated salinity on soil structure, nutrient availability and microbial activity. Additionally, exploring the suitability of TTWW for irrigating other salt-tolerant crops and its compatibility with diverse irrigation systems could offer further insights into optimizing agricultural water usage efficiency.

This research contributes significantly to several Sustainable Development Goals (SDGs). The development of an energy-efficient pilot plant for the treatment of highly saline TWW addresses SDG 6 (Clean Water and Sanitation) by promoting efficient water management practices and improving access to clean water resources. This work also aligns with SDG 14 (Life Below Water), by mitigating the discharge of pollutants into marine ecosystems, and SDG 7 (Affordable and Clean Energy), by prioritizing energy efficiency in wastewater treatment processes. Additionally, the reuse of treated TWW in irrigation supports SDG 2 (Zero Hunger) by ensuring sustainable agricultural practices through informed water management strategies.

In conclusion, treating and reusing saline TWW for irrigating salt-tolerant crops offers a sustainable solution for wastewater management and agricultural production in water-stressed regions. Continued research will enhance the resilience of industrial and agricultural sectors to water scarcity and environmental degradation, fostering sustainable development, especially in arid and semi-arid regions.

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## CONFLICT OF INTEREST STATEMENT

The authors declare no conflict of interest concerning this article's research, authorship and publication.

## DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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