



Assessment of impacts of industrial effluents on physico-chemical and microbiological qualities of irrigation water of the Fez Rriver, Morocco

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Abstract Global water consumption has grown twice as fast as the population. Wastewater is therefore a valuable and renewable source and provides additional water for priority uses. Wastewater can also be a source of pollution; thus, its physico-chemical and biological compositions can present major risks to the environment and human health. The objective of this study was to assess the status of irrigation waters in terms of salinization, accumulation

of metallic elements, and microbiological contamination by parasites and pathogenic bacteria. The study focused on the surface water of Oued Fès used for irrigation located downstream of the industrial zone of Doukkarat and upstream of the industrial zone of Ain Noukbi (wastewater) before the confluence with the Oued Sebou, as well as on the treated wastewater of the wastewater treatment plant. The physico-chemical and microbiological analyses were carried

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out in two periods: summer and winter. Metals were analyzed by ICP-AES. The chemical and bacteriological quality of the wastewater and treated wastewater was found to be poor. These were characterized by organic pollution, including biodegradable pollutants, while upstream the organic residues were not biodegradable. COD, BOD₅, Kjeldahl nitrogen, as well as chloride ion (Cl⁻) are above the standard values. The highest concentrations of Cd, at 850 µg/l, Cu, at 690 µg/l and Mn, at 470 µg/l, largely exceed the international standards and requirements. In addition to fecal contamination, characterized by total coliforms and thermo-tolerant coliforms, other pathogens were present, including helminth eggs, both in the wastewater and in the treated wastewater. Other pathogens, such as *Vibrio cholera*, were found at all three sites whether in winter or summer, with the exception of the downstream of Oued Fez in winter. As for *Salmonella*, it was present in treated wastewater during the winter only. The water used for irrigation upstream of Oued Fez and the treated wastewater have poor to very poor quality. Therefore, for a better use of these waters, it is necessary to ensure their regular treatment in order to minimize the impacts on the environment and human health.

Keywords Irrigation water quality · Wastewater · Treated wastewater · Pathogenic bacteria · Metals · Fez · Morocco

Introduction

Demographic growth and economic development are putting pressure on renewable, yet limited water resources. It is estimated that by 2025, 1.8 billion people will live in countries or regions with less than 500 m³ of renewable water per capita per year (FAO, 2007; Hamoda, 2004). This means that current usage of water in the world will exceed capacities of water resources to be renewed (Remon,

2018). Due to the Mediterranean region being known for its arid to semi-arid climate, scarcity of water will be a major issue in those countries. Almost all the accessible freshwater resources in the region have already been mobilized (Hamdy, 2002). Therefore, to meet increased demand, it is natural to turn to unconventional sources of water. To preserve good-quality water resources for drinking water, use of drainage water, of marginally high salinity or wastewater (WW) can be used for irrigation of some crops (FAO, 2007; Qadir et al., 2007). Among the areas of potential water reuse, irrigation is the most promising sector. In fact, agriculture consumes more than 70% of water resources, especially in developing countries (Qadir et al., 2007). Currently, untreated water is used to irrigate 10% of the world's crops (Papaiacovou, 2001; Scott et al., 2004). Furthermore, wastewater can be a valuable source of fertilizers, particularly phosphorus and nitrogen, but also organic matter, yet it can also be contaminated and thus a source of pollution. Its content of organic and metallic trace elements and pathogens, as well as its high nitrogen content, can be a threat to the environment and human health (Belaid, 2010; Jampani et al., 2020). Morocco has historically and is currently suffering from scarcity of water. With changes in climate, there have been a general increase in severe natural phenomena and a significant reduction in precipitation in some regions, such as North Africa (Azzaoui et al., 2002). In addition, agriculture represents a key sector of the national economy, which in 2016 contributed nearly 13.6% of the gross domestic product (Bouchetara et al., 2021), and employs 36.6% of the active population. In response to these effects, one of the measures to meet the needs is the use of treated wastewater (TWA) in irrigation. The volume of wastewater discharged by Moroccan cities is estimated at nearly 550 million m³ per year, 45% of which is treated by 117 plants (Er-Raioui et al., 2012). The National Water Strategy plans to reuse 31% of its WW by 2030. According to the Ministry of Equipment, Transport, Logistics, and Water (MEE), the volume of purified wastewater mobilized for reuse by the end of 2020 was to be approximately 71 m³, of which nearly 51% was to be dedicated for watering golf courses and green spaces and 17% for industrial use (Diao, 2021). According to the Delegation of Trade and Industry

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of Fez in 2015, the city of Fez represented the core of economic activity in the Fez-Boulemane Region. It has six industrial zones with nearly 614 industrial establishments, whose main activities are food processing, textiles, leather and metallurgy (Mahboubi & Benyacoub, 2020). The agricultural use of the WW in the Oued Fez and Sebou in the region of Fez is used primarily for market gardening (Derwich et al., 2008). This usage, according to the Ministry of Commerce and Industry (Perrin et al., 2014), is accompanied by health risks, assessment of which requires knowledge of these physico-chemical and microbiological characteristics (Idrissi et al., 2015). The present study was conducted to evaluate effects of irrigation with WW and TWA in Fez. The objectives of this work are to examine the degrees of pollution and the quality of water used in irrigation through the analysis of different physico-chemical parameters of metallic elements and microbiological contamination of parasites and pathogenic bacteria.

Material and methods

Description of the study sites

Surveillance trips allowed identification of sources of water, as well as the immediate area around sources of water used for irrigation in the Oued Fez watershed upstream and downstream before its meeting with Oued Sebou and irrigated land by TWA at the exit of the Water Treatment Plant (STEP) (Fig. 1). Types of irrigation used is surface irrigation for the 3 agricultural lands were also determined. For each source, two samples were collected, one during each period. Samples were taken during the low-water period corresponding to the dry season of August 2019 and then during the flood period corresponding to January 2020 (Table 1). Samples of water intended for the physico-chemical analyses were taken in PVC bottles that had been carefully cleaned, and rinsed with distilled water to a capacity of 2 L.

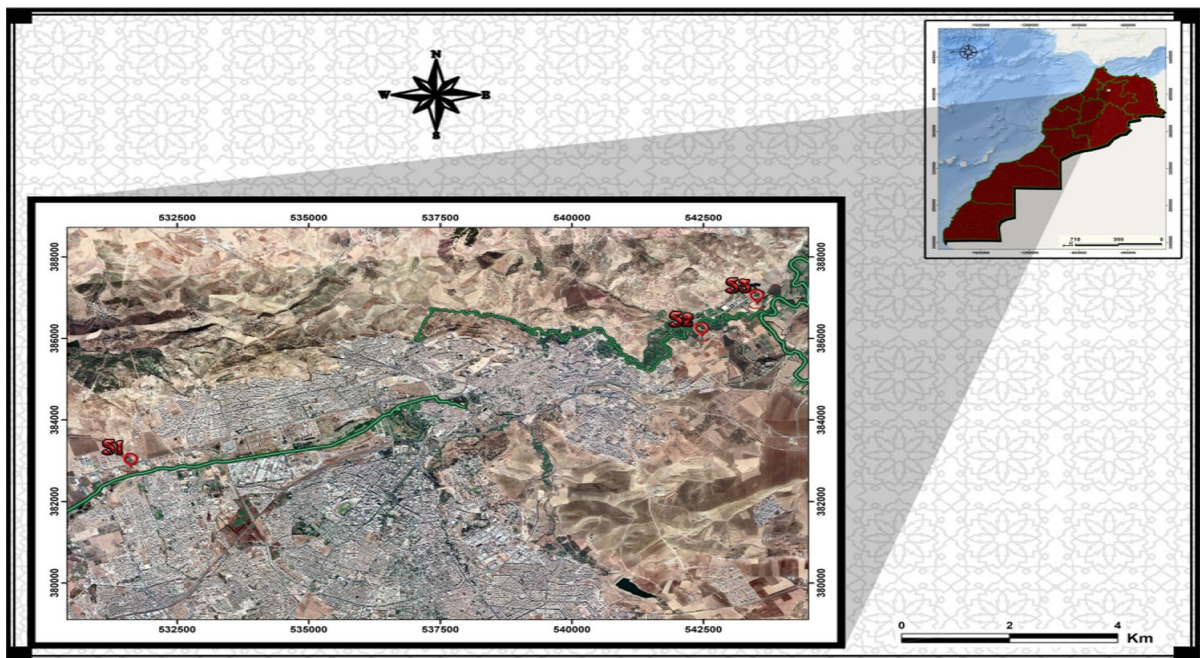


Fig. 1 Locations of sampling stations as well as locations of pumps used for. S1: Irrigation water Oued Fez located before the industrial zone Doukkarat. S2: Irrigation water Oued Fez

located after the industrial zone Ain Nokebi. S3: Irrigation water output from the STEP located after the STEP before the meeting of Oued Sebou

Table 1 Descriptions of sampling sites

Station Name and Address	Geographical coordinates		Human pressure
	Latitude N	Longitude W	
S1 Irrigation water Oued Fez located before the industrial zone Doukkarat	34°02'35.9"	5°03'19.3"	Urban/agricultural
S2 Irrigation water Oued Fez located after the industrial zone Ain Nokebi	34°04'21.1"	4°56'18.4"	Agricultural
S3 Irrigation water output from the STEP located after the STEP before the meeting of Oued Sebou	34°04'49.0"	4°55'37.0"	Agricultural

Water sampling and analysis

During sampling, hand-held bottles were immersed to a depth of 20 cm with the neck pointing against the current (Rodier, 2009). Bottles filled with water were removed from the water body and sealed. Samples of water for bacteriological analyses were taken using borosilicate glass vials washed thoroughly with distilled water. Cleaned and rinsed vials were sterilized in an autoclave at 120 °C at 120 kg/cm² for 30 min. Labeled samples were placed in a cooler with ice and transported to the laboratory. In situ, measurement of physico-chemical parameters included pH, electrical conductivity (EC), total dissolved solids (TDS), and temperature (T). The pH was measured with a pH meter, while the EC and temperature were measured with a conductivity meter. In the laboratory, the analyses carried out on the irrigation water are essentially based on the physico-chemical and bacteriological components that are characteristic of pollution. The physico-chemical parameters were determined according to the following chemical methods: chloride, Cl⁻: The volumetric method of MOHR where chlorides are totally precipitated by silver (AgCl₂) was used. The end of the titration is marked by the chromate indicator (K₂CrO₄), which forms a red precipitate (AgCrO₄) with silver excess.

Chemical oxygen demand (COD) and five-day biological oxygen demand (BOD₅), representing the

proportion of biodegradable organic matter, were conducted in the laboratory according to the analytical methods recommended by Moroccan standards and by Rodier et al (2009). Kjeldahl nitrogen (KN) is the sum of organic nitrogen, ammonia (NH₃), and ammonium (NH₄⁺). The determination of KN consists of two phases: digestion and distillation, according to the analytical method of Rodier (1996).

Metals, Cd, Cr, Cu, Ni, Pd, Fe, Mn, and Zn, were quantified by use of inductively coupled plasma atomic emission spectrometry (ICP-AES) at the laboratory of the Innovation Campus of Mohammed Ben Abdallah University of Fez. This device works in simultaneous mode. The experimental parameters are power: 1,3 kW; plasma gas flow: 15 L/min; auxiliary gas flow rate: 1.5 L/min; nebulization pressure: 200 kPa; stabilization time: 20 s; reading time per replica: 5 s; pump speed: 15 rpm end flush time: 30 s. The standard solution used is Merck's high-quality Certipur®, single and multi-element (1000 mg/L). The Certipur® standards used are traceable to NIST SRM.

The microbiological parameters tested included total coliforms (TC), thermos-tolerant coliforms (TTC) *E. coli*, *Salmonella*, and *Vibrio cholerae* (Vc). Regarding the parasites, we analyzed the eggs of helminths (Table 2).

For helminth eggs (HE), identification was made from the pellet obtained according to the Rodier

Table 2 Methods for identification and quantification of bacteria

Microorganisms	Culture medium	Incubation time	References
Total coliforms	Violet Red Bile Lactose Agar (VRBL) or Mac Conkey	24 h at 37 °C	NM ISO 9308–1
Thermo-tolerant coliforms	Violet Red Bile Lactose Agar (VRBL) or Mac Conkey	24 h at 44 °C	NM ISO 9308–1
<i>Salmonella</i>	Salmonella chromogenic agar	24 h at 37 °C	NM ISO 19250
<i>Chloric vibrio</i>	The TCBS environment	24 h at 37 °C	NM 03.7.051

experimental protocol (1996). The enumeration of the eggs was done with the help of the MacMaster slide under a photonic microscope.

Statistical analyses

The statistical analyses performed on the parameters are both univariate (correlation tests) and multivariate (similarity tests and principal component analysis) to help determine different correlations between the physico-chemical parameters. The data were checked to see if it met the requirements of normality and homogeneity of variances required to apply parametric statistics.

The statistical analyses were processed by IBM SPSS statistical version 27 software (University of New Haven; West Haven, CT, USA).

Data for water physico-chemical and bacterial pollutants in were listed and analyzed. All experiments were performed in triplicate per sampling sites.

Before conducting PCA, a Shapiro–Wilk test was used to help confirm the normality and homogeneity of variances as well as to emphasize variations or similarities in the composition between sampling sites (temporal and spatial variability). The eigenvalues were reported, and the amount of variances was determined. Once the set of eigenvalues determined, possible relationships between the 16 parameters were ascertained by correlation coefficients. The number of principal components to be considered and a graphical representation of the results were generated.

Results and discussion

The status and trends of the physico-chemical and microbiological parameters were investigated by use of spatio-temporal variation in two steps. The first was temporal trends in which means of each parameter were calculated across locations for the summer and winter periods, and then calculating annual mean values. The second evaluated spatial trends of these same parameters among stations.

Evaluation of physico-chemical properties

Physico-chemical parameters varied by season and location (Tables 3 and 4). Temperatures of irrigation

Table 3 Spatial and temporal evolution of the physico-chemical parameters of different stations

Parameters	T	pH	EC (µs/cm)	TDS (mg/l)	COD (mg/l)	BOD ₅ (mg/l)	COD/BOD ₅	Nk (mg/l)	Cl ⁻ (mg/l)
S1	Wet season	9.12 ± 0.4	7.63 ± 0.25	930 ± 28.35	595.35 ± 0.39	34.43 ± 0.5	29 ± 0.81	22.04 ± 0.04	163.27 ± 3.46
	Dry period	17.37 ± 0.5	7.02 ± 0.12	594.67 ± 6.50	380.59 ± 0.01	105.67 ± 1.01	34.67 ± 0.47	20.97 ± 0.025	113.57 ± 0.68
Annual M.V	Wet season	13.25 ± 4.12	7.33 ± 0.43	762.33 ± 237.11	487.97 ± 107.38	70.05 ± 50.37	31.83 ± 2.83	21.50 ± 0.76	138.42 ± 35.14
	Dry period	9.61 ± 0.08	7.68 ± 0.07	994.67 ± 10.79	636.63 ± 0.05	201.63 ± 0.96	47.33 ± 1.25	42.05 ± 0.02	170.47 ± 0.47
Annual M.V	Wet season	23.47 ± 0.43	7.49 ± 0.05	2020.67 ± 4.73	1293.22 ± 0.02	652.77 ± 2.20	237 ± 0.82	53.78 ± 0.02	504.1 ± 1.05
	Dry period	16.53 ± 6.93	7.59 ± 0.47	1507.67 ± 725.4	964.92 ± 328.29	427.2 ± 319	142.17 ± 94.83	47.92 ± 0.189	337.27 ± 235.9
S3	Wet season	12.43 ± 0.32	7.38 ± 0.34	1830 ± 11.53	1171.3 ± 0.22	691.27 ± 3.11	81.67 ± 1.25	77.09 ± 0.01	426.67 ± 3.06
	Dry period	23.43 ± 0.35	7.50 ± 0.08	1333.33 ± 26.65	853.33 ± 0.02	441.6 ± 0.62	146.33 ± 0.94	64.06 ± 0.017	426.87 ± 0.80
Annual M.V	Wet season	17.93 ± 5.50	7.77 ± 0.13	1581.67 ± 351.2	1012.31 ± 158.99	566.43 ± 176.54	114 ± 32.33	70.58 ± 0.73	427.77 ± 0.14
	Dry period	35	6.5–8.4	12,000	450	250	120	30	350
Moroccan standards	–	6.5–8.4	< 3 ds/m	< 2000	–	–	–	30	> 10 meq/l (mg/l) equivalent weigh = meq/l)
FAO (1994)	–	6.5–8.4	< 3 ds/m	< 2000	–	–	–	30	> 10 meq/l (mg/l) equivalent weigh = meq/l)

Table 4 Concentrations of metals at each location

Heavy metals (µg/l)		Cd	Cr	Cu	Ni	Pd	Fe	Mn	Zn
S1	Wet season	12±0.0001	40±0.001	30±0.00008	10±0.0004	10±0.001	10±0.001	10±0.001	20±0.02
	Dry period	430±0.01	180±0.005	240±0.007	610±0.01	310±0.004	550±0.001	220±0.002	260±0.01
	Annual M.V	220±0.42	110±0.42	135±0.42	310±0.12	160±0.19	280±0.56	115±0.3	140±0.52
S2	Wet season	10±0.0002	50±0.007	690±0.01	200±0.003	10±0.001	50±0.001	30±0.001	200±0.01
	Dry period	850±0.004	280±0.008	400±0.02	1130±0.01	610±0.01	1050±0.003	420±0.21	30±0.001
	Annual M.V	430±0.21	165±0.21	546±0.21	670±0.06	310±0.13	550±0.2	225±0.15	115±0.25
S3	Wet season	12±0.0002	40±0.0008	30±0.002	10±0.001	10±0.001	10±0.0008	10±0.001	10±0.001
	Dry period	850±0.08	370±0.01	690±0.01	1160±0.04	620±0.02	1410±0.01	470±0.02	100±0.01
	Annual M.V	430±0.42	210±0.42	360±0.17	585±0.58	315±0.31	710±0.7	240±0.23	55±0.01
Moroccan standards		10	1000	200	200	5000	5000	200	2000
FAO (1994)		<10	<100	<200	<200	<5000	<5000	<200	<2000

water followed a seasonal pattern and varied between 9.12 and 12.43 °C during winter and 17.37 and 23.43 °C during summer. The temperature tends to increase as we move away from the upstream of Oued Fez. That is to say, a warming of 3.31 °C in winter and 6.06 °C in summer due to anthropic developments tend to modify the thermal regime of the rivers. These variations can directly influence the lives of aquatic organisms (Eldridge, 1967). Recorded pH values ranged from 7.02 to 7.68 and are generally alkaline to somewhat neutral. A slight difference was observed from upstream to downstream.

Natural environmental variables, such as vegetation cover, the nature of the bedrock and soil, and human activities, such as pollution, influence the pH (Brémond & Vuichard, 1973; Dussart & Fauré-Fremiet, 1966). It is noticed to reduce when water levels are low and evaporation is high. On the other hand, pH increases for TWA during dry periods since the flow does not depend on geothermal conditions but on human activities.

EC is one of the physical parameters that allows verification of amounts of dissolved salts in water (Rodier, 1996) and the presence of pollution in water (Ghazali & Zaid, 2013; Rezouki et al., 2021a, 2021b). It is a better parameter for water geochemical differentiation (Merghem et al., 2016). The EC

of the waters of Oued Fez presents significant values that vary strongly between upstream 594.67 µs/cm and downstream wastewater 2020.67 µs/cm in the dry period. These values are comparable to those found in Wadi Zegzel (Morocco) by Derfoufi et al. (2019). On the other hand, they are higher than those encountered in these same waters in 2007 (Derwich et al., 2008). The increased EC during the hot period might be related on the one hand to the low flow of the Oued, which leads to increased mineral salt concentrations, and on the other hand to high atmospheric evaporation (Lamri & Belghyti, 2011).

In summer the EC of 1.3×10^3 (µs/cm) in the TWA was less than that observed during winter. Salinity was calculated from electrical conductivity. Salinities of all the samples were classified as having minimal to moderate restriction which meets the requirements of water reuse for irrigation (EC < 3ds/m). These results are similar to those found in Wadi Zegzel (Morocco) by Derfoufi and in Zelfana in Algeria (Benslama, 2021). Excessive concentrations of salt in freshwater or soil negatively affect agricultural production and adversely affect groundwater. Salinity is related to increased mobility and bioavailability of trace metals in soil solution. Once salinized, soil is

difficult to remediate (Acosta et al., 2011; Du Laing et al., 2008).

Chemical oxygen demand (COD) is used to estimate concentrations of organic or mineral matter, dissolved or suspended in water that can be oxidized, through the amount of oxygen required for their total chemical oxidation (Rodier, 1996). Concentrations of COD observed ranged from 34 to 6.9×10^2 mg/L, with a maximum annual mean of 5.7×10^2 mg/L, which was observed for TWA. The wastewater is of very poor quality since it exceeds 250 mg/l, which is the Moroccan standard (Official Buletin, 2002). The least annual, mean COD observed was 70 mg/L, observed upstream of Oued Fez. Waters at that location were less polluted because they were upstream of the industrial areas of Fez. These results are more significant in comparison with the results found in surface waters of Bamenda, Cameroon (Mufur et al., 2021).

Five-day biological oxygen demand (BOD_5) is an indicator of biodegradable organic matter and is also assessed to determine the amount of oxygen required to stabilize domestic and industrial waste so that once discharged it does not deplete oxygen in surface waters, which can cause fish to be killed (Derfoufi et al., 2019; Gupta et al., 2017; Rezouki et al., 2021a, 2021b). In summer, WW to be used for irrigation at Oued Fez was rich in organic matter with BOD_5 in the range of 2.4×10^2 mg/l. The BOD_5 of water at TWA was less, with a mean of 1.5×10^2 mg/l. For the flood season, the respective results are 4.7×10^1 mg/l and 8.2×10^1 mg/l. Variations observed among samples are significant and suggest a remarkable modification in the market gardening activity lately. These waters exhibited strong pollution by organic elements. This organic pollution is greater during the dry season, compared to the rainy season. These findings are also in agreement with data from the surface waters of Wadi Zegzel (2019) and the waters of the Narmada River, Madhya Pradesh, India (2017). Leaching of organic fertilizers and the heavy use of industrial WW in the city of Fez are the main causes of this organic pollution. The presence of great concentrations of organic elements constitutes an important contribution of structural elements to the soil during irrigation. The COD/ BOD_5 ratio is important for determining appropriate treatments of effluents. Lesser COD/ BOD_5 ratios imply the presence of a large proportion of biodegradable elements, for which biological

treatment is appropriate. Conversely, greater values of this ratio indicate that a large part of the organic matter is not biodegradable and, in this case, it is preferable to consider physico-chemical treatments. From the COD/ BOD_5 , it can be deduced whether wastewater should be discharged directly into receiving environments. In fact, it has the characteristics of domestic waste (COD/ BOD_5 ratio less than 3). Values of this ratio are an indication of presence of pollutants with little or no biodegradability that should be treated before release into the environment. Aquatic environments poses assimilative capacities so that they can accommodate releases of defined amounts of BOD_5 over defined periods of time. According to NBM, the annual average value of the COD/ BOD_5 ratio upstream of Oued Fez is 2.2, which is less than the value of 3.0 defined by the WW as being acceptable. Thus, it can be concluded that even though urban waste has a large organic load, it is easily biodegradable. The examination of this ratio underlines the biodegradable characteristics of wastewater mixed with the discharges of the municipal slaughterhouse and the regional hospital of the city, for which a biological treatment seems quite suitable. The COD/ BOD_5 ratio of the downstream wastewater and TWA was greater than 3. Therefore, a large part of the organic materials are not biodegradable since these waters have accumulated different industrial pollutants, and, in this case, it is preferable to consider physico-chemical treatment.

Total nitrogen characterizes the total nitrogen pollution of an effluent. In this study, concentrations of Kjeldahl nitrogen varied among locations and between seasons. Downstream irrigation WW had a greater concentration of 5.4×10^1 mg/l during in summer and a lesser concentration of 4.2×10^1 mg/l in winter, but these concentrations were still greater than the water quality standard of 30 mg/l. Results for TWA fluctuated, with an annual average of 6.9×10^1 mg/l, which is influenced by the large concentration of 1.3×10^2 mg/l, observed during winter.

According to the Moroccan standards of water quality for irrigation, chlorides Cl^- should not exceed 3.5×10^2 mg/l for surface irrigation. The WW had a concentration of 5.0×10^2 mg/l during summer, which exceeded the water quality criterion. That site was characterized by strong mineralization during summer due to reduced water inflow and higher temperature, which cause the water to become enriched

in chlorides (Karrouch et al., 2017). Values of TWA were the same in winter and summer 4.3×10^4 mg/l. These results are consistent with waters from the Bystrzyca River in Poland (Tomczyk & Wiatkowski, 2021) and data from Karrouch (2017) downstream of the Boufekrane River in Meknes, Morocco.

Spatial and temporal variation in metals

Metals in aquatic environments can come from a variety of sources, including either natural sources or from human activities. Metals are naturally present in soils, with some being major constituents, such as Al or important for structures of minerals, such as Fe and Mn (Baize & Sterckeman, 2001; Horckmans et al., 2005). Metals can be released by industrial activities where they are used as reagents for surface treatments, re and reaction intermediates in agriculture where they are used as phytosanitary agents. Urban wastewater is composed of a mixture of domestic waste, industrial wastewater, and runoff water. Metals in runoff come from the atmospheric deposition, but also from corrosion of runoff surfaces, such as roofs and gutters (Gromaire et al., 2001). In this study, eight trace elements, Cd, Cr, Cu, Ni, Pb, Fe, Mn, and Zn, were investigated (Table 4), whose highest average concentrations are, respectively, 430 µg/L, 200 µg/L, 360 µg/L, 550 µg/L, 320 µg/L, 710 µg/L, 240 µg/L, and 140 µg/L. The average concentrations of these trace elements were higher in summer than in winter. The concentrations of Cd and Mn exceeded the Moroccan standards and the FAO guidelines. The concentration of Cd is also high according to Sussy Sayo

(2020) in waters downstream of Embu, Kenya (Sayo et al., 2020). On the other hand, Cr values are out of limit according to FAO (< 100 µg/l) and those at S2 and S3 waters 170 µg/l and 210 µg/l. Some of these metals are of geological and anthropic origin and reach the fluvial system through hydro-climatic erosion. Indeed, the environment of Oued Fès is strongly alkaline whose sediments are of silicate clay and carbonate nature with a texture with variable granulometry (clayey, silty, and sandy) (Bissassa, 2016). According to the studies done by Bissassa (2016) on the rate of heavy metals out of the norm found in the sediments downstream of Oued Fez are $Zn > Cu > Pb$ with the decreasing values of $700 \text{ mg/kg} > 500 \text{ mg/kg} > 250 \text{ mg/kg}$. The mobility of heavy metals in sediments is highly influenced by their composition in inorganic and organic constituents. The main metal-bearing phases are organic matter and clays with surface and loading properties that give them significant adsorption capacities (Tables 5 and 6).

Table 5 Indicator microorganisms, total coliforms, thermo-tolerant coliforms and parasites, helminth eggs

		Total coliforms (UFC/100 ml)	Thermo-Tolerant Coliforms (UFC/100 ml)	Helminth eggs (Euf/l)
S1	Wet season	70,063.33	2224.666667	0
	Dry period	1,300,156.66	71,030	24.67
	Annual M.V	685,110	36,800	12.33
S2	Wet season	1,100,110	480,046.6667	50
	Dry period	880,083.33	150,036.6667	32
	Annual M.V	990,055	315,041.6667	41
S3	Wet season	20,000,156.67	2,000,046.67	479.67
	Dry period	2,400,060	1,360,066.667	76
	Annual M.V	11,200,108.33	1,680,056.667	254.83

Table 6 Presence or absence of pathogenic microorganisms, *Salmonella* and *Vibrio cholerae*

		<i>Cholera Vibrio</i>	<i>Salmonella</i>
S1	Wet season	Absent	Absent
	Dry period	Presence	Absent
S2	Wet season	Presence	Absent
	Dry period	Presence	Absent
S3	Wet season	Presence	Presence
	Dry period	Presence	Absent

Indicator pathogens

All of the pathogens studied exceeded Moroccan standards of water quality for irrigation, which requires maximum total coliforms of 1.0×10^3 CFU/100 mL. In TWA, the mean, annual concentration of total coliforms was 1.1×10^7 CFU/100 mL, followed by the WW with of 9.9×10^5 CFU/100 mL. This difference can be explained by the large quantity of water collected from the city of Fez which contains large amounts of feces. The maximum mean annual of numbers of thermos-tolerant coliforms was observed in TWA being 1.7×10^6 CFU/100 mL. The samples are more loaded with total coliforms in the dry period than in the rainy period. This characteristic of the WW has also been noted by other authors in Africa (Oulibaly-Kalpy et al., 2017). Indeed, contamination of these reservoirs was superposable to that in the urban WW (Ouali et al., 2013). Thus, the source of the contamination in the reservoir was driven by that in wastewater.

Mean annual numbers of helminth eggs in WW were approximately 41 eggs/l, while that for TWA were 2.5×10^2 eggs/l. Parasitic helminth eggs in urban wastewater were associated with the number of inhabitants connected to the STEP. During winter, water upstream of Oued Fès did not contain helminth eggs, but during summer 2.5×10^1 eggs/l were observed. Because of their resistance and persistence in the environment, the sanitary risk posed by helminth eggs during reuse of WW in agriculture was significant (Derwich et al., 2008; Hamaidi & Kais, 2016). Waters from upstream of the discharges of the city of Fez partially met criteria of quality standards for water intended for irrigation. However, waters from regions located downstream of the city and the confluence of Oued Fez were most polluted with microbes and parasites. Therefore, farmers in the Oued Sebou watershed would be exposed to bacterial and parasitic diseases.

Pathogenic microorganisms

Morocco does not tolerate any *Salmonella* detectable in 5 L or *Cholera vibrio* contamination in 450 ml of irrigation water. During summer, *C. vibrio* was present at all three locations. During winter, *C. vibrio* was absent in the upstream Oued

Fès WTs, but present downstream in WW and TWA. These results are not consistent with those found in 2010, 2011, and 2012 in Oued Fes by Ouali et al. (2014). Alternatively, *Salmonella* was absent from all three sites except for TWA during winter. Pathogenic bacteria of the genus *Salmonella* were not detected in the studies of Hamaidi and Kais (2016), while the results of Ndiayee al. (2011) showed that 35% of irrigation water was contaminated with *Salmonella* spp. between the two types of water used for irrigation (groundwater and wastewater). This absence has been reported in other studies of WW, despite the presence of a high fecal bacterial load. Studies of effect of irrigation methods on the level of contamination of vegetables showed that even for highly contaminated irrigation water, drip or furrow methods significantly reduced the risk of crop contamination (Hamaidi & Kais, 2016; Ndiaye et al., 2011).

Statistical data analysis

Normality test

The Shapiro–Wilk test is more appropriate for small sample sizes. This test showed that all parameters studied met the requirements for normality. The results are summarized in Table 7.

Principal component analysis (PCA) test

The PCA test was applied to identify the crucial parameters that characterize the water quality of the studied stations. These components define a space of reduced dimensions in which are projected the initial variables accounting for the maximum information. The results of the physico-chemical as well as microbiological data by PCA with the most significant correlations are presented in Tables 8, 9 as well as depicted in Fig. 2.

Table 8 represents the eigen values, variability, and accumulation.

From Table 8, the first two components were extracted and utilized for the analysis. Two maximum components are chosen. The contributions of different parameters in the expression of the two first components are, respectively, 46.029% and 37.320%. This represented a total explained variance of 83.349%

Table 7 Normality test results

	Normality tests					
	Kolmogorov–Smirnov ^a			Shapiro–Wilk		
	Statistics	ddl	Sig	Statistics	ddl	Sig
Pd	.294	6	.115	.796	6	.054
T	.208	6	.200*	.858	6	.182
pH	.234	6	.200*	.879	6	.265
CE	.200	6	.200*	.942	6	.674
TDS	.232	6	.200*	.903	6	.391
COD	.195	6	.200*	.885	6	.292
BOD	.234	6	.200*	.849	6	.155
NK	.195	6	.200*	.892	6	.330
Cl ⁻	.293	6	.116	.814	6	.078
Cd	.268	6	.200*	.835	6	.118
Cr	.282	6	.148	.849	6	.153
Cu	.207	6	.200*	.864	6	.202
Ni	.226	6	.200*	.841	6	.133
Fe	.279	6	.158	.842	6	.136
Mn	.281	6	.152	.822	6	.091
Zn	.258	6	.200*	.861	6	.192

(Table 8). The maximum of the total inertia is accumulated by the plan formed by the factorial axes component1 × component2 (Fig. 2).

Table 8 Eigen values and percentage contribution of the 16 variables

Component	Initial eigen values		
	Total	% of variance	% Cumulative
1	7.365	46.029	46.029
2	5.971	37.320	83.349
3	1.706	10.663	94.012
4	.640	3.998	98.009
5	.318	1.991	100.000
6	8.291E-16	5.182E-15	100.000
7	6.376E-16	3.985E-15	100.000
8	4.207E-16	2.630E-15	100.000
9	3.662E-16	2.289E-15	100.000
10	3.902E-17	2.439E-16	100.000
11	-5.610E-18	-3.507E-17	100.000
12	-1.462E-16	-9.135E-16	100.000
13	-4.013E-16	-2.508E-15	100.000
14	-1.028E-15	-6.422E-15	100.000
15	-1.553E-15	-9.709E-15	100.000
16	-1.785E-15	-1.116E-14	100.000

Table 9 represents the correlation matrix. In Table 9, it is shown that the following variables have a strong positive correlation coefficient between chemical parameters: TDS showed a significant correlation between EC conductivity (0.994) and also COD (0.964); the latter itself also correlated strongly with Cl⁻ (0.945). The results of the analysis of metals exhibited strong correlations, such as: Fe*Cr (0.999); Mn*Ni (0.994) and Mn* Pd (0.996). These data occur because of the interaction between different metals.

A negative correlation coefficient was observed between the temperature and the metal variables such as: Cd - (0.831); Ni - (0.802) and Mn - (0.805). According to Fonseca et al. (2013), water temperature is a factor that changes the availability of pollutants.

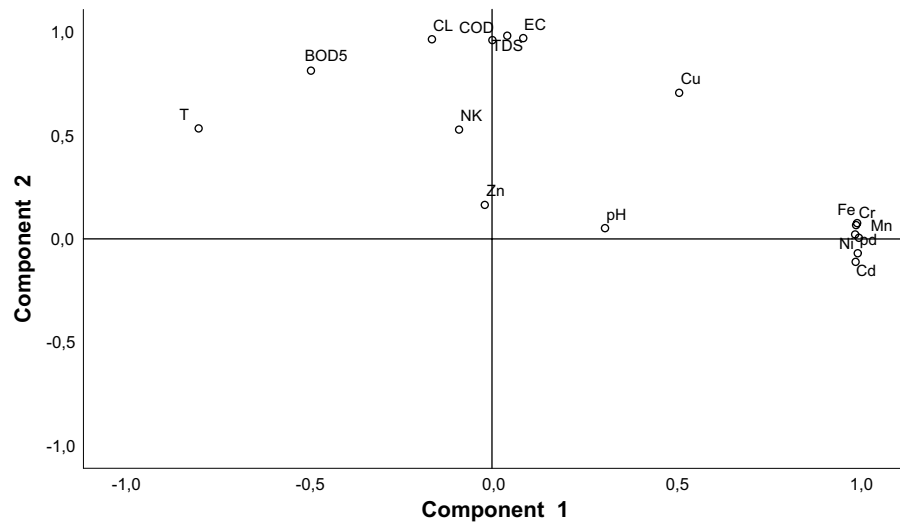
These correlations reflect the influence of the parameters between them, impacting the quality of the water used in irrigation. Figure 2 represents the correlation between different parameters physico-chemical and microbiological on the factorial plane.

The analysis of the distribution of the parameters in the component 1 × component 2 plans was conducted to identify trends, correlations, and phenomena that may influence the distribution of chemical and microbiological elements in irrigation waters.

Table 9 Mmtrix of pair-wise correlations between 16 variables

T	pH	EC	TDS	COD	BOD ₅	KN	Cl ⁻	Cd	Cr	Cu	Ni	Pd	Fe	Mn	Zn
T	1.000														
pH	-.308	1.000													
EC	.402	.245	1.000												
TDS	.447	.145	.994	1.000											
COD	.473	.065	.955	.964	1.000										
BOD ₅	.807	.139	.797	.807	.750	1.000									
KN	.147	.517	.643	.664	.570	.559	1.000								
Cl ⁻	.663	.107	.935	.945	.878	.576	-.164	1.000							
Cd	-.831	.330	-.029	-.107	-.576	.021	-.092	.971	1.000						
Cr	-.775	.316	.116	.099	-.447	.461	.531	.385	.540	1.000					
Cu	-.115	.268	.781	.646	.402	.004	-.141	.971	.978	.571	1.000				
Ni	-.802	.425	.083	.015	-.425	-.098	-.218	.996	.980	.451	.988	1.000			
Pd	-.831	.369	.026	-.025	-.530	-.007	-.084	.973	.999	.544	.978	.982	1.000		
Fe	-.763	.302	.162	.099	-.441	-.038	-.152	.987	.992	.519	.994	.996	.993	1.000	
Mn	-.805	.364	.103	.054	-.474	.849	.124	-.091	.057	.428	.095	-.017	.024	.029	1.000
Zn	-.166	.469	.330	.283	.270										

Fig. 2 Correlation graph of physico-chemical and microbiological parameters on the factorial plane



At the level of the plane formed by component 1 and component 12 (Fig. 2), there is a set of variables such as Mn, Cd, Fe, and Cr which are correlated on the component 2 axis with variability of 37.053%. There is also a correlation between the variables Cl^- , TDS, COD, and EC on the component 1 axis, with variability of 39.190%.

Conclusion

As the population increases and water supplies decrease due to climate change, water reuse is becoming more common. In particular, the use of wastewater for irrigation is becoming more common, especially in times of drought. Wastewater not only provides needed moisture, but also additional nutrition, but as the results of the study presented here have shown, there are risks of environmental and public health impacts (FAO, 2003). The physical–chemical and microbiological quality of wastewater used for crop irrigation does not always meet the criteria during the winter and summer periods. The irrigation waters of the stations located downstream of the Doukkarat industrial zone in the city of Fez and upstream of the Ain Noukbi industrial zone before the confluence with the Oued Sebou are chemically and bacteriologically polluted. They are characterized as biodegradable organic pollution because the COD/BOD₅ ratio is less than or equal to 3. It is contrary to the TWA which contains a non-biodegradable

pollution since the COD/BOD₅ ratio is higher than 3. The data of nitrogen NK and chloride Cl^- are out of norms at the level of the EU of Oued Fès and the TWA in both seasons. The concentrations of metals exceeded the quality criteria for irrigation water, especially for Ni, Cd, Cu, and Mn found in the WW and TWA. In addition, contamination with fecal pollution indicator germs, total coliforms and thermotolerant coliforms, as well as the presence of helminth eggs, contributed to the risks posed by the WW and TWA. Among the pathogenic microorganisms, vibrio cholera was present in all three sites and in both the winter and summer periods, with the exception of the waters of Oued Fez located upstream in winter. For salmonella, this pathogen was present in winter only in TWA. If these waters are to be used for agricultural irrigation, they must be improved and protected from further contamination. In the short term, practices such as crop selection and type and frequency of irrigation may allow for safe use of these unconventional waters, but monitoring must be done beforehand and possible treatment applied if necessary. In the medium term, due to bacterial contamination, additional treatment of wastewater in Oued Fez should be considered before using it in agriculture.

Author contributions FJ carried out the sampling and the physicochemical and microbiological analyses with the support of BAMRS, MT, ASM, MB, FK, MAMA-S, and JPG. She supervised the data and statistical calculations. LA and EN supervised the project. MAMA-S and JPG contributed to writing the original draft and editing the final version. All authors

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Data availability All data pertinent to this study are available upon request to the corresponding author.

Declarations

Conflict of interest Authors declare no conflict of interest.

Consent for Publication Not applicable as this is not a clinical study.

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