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Water quality for agricultural irrigation produced by two municipal sewage treatment plants in Santiago Island-Cape Verde: Assessment of chemical parameters and pharmaceutical residues

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ABSTRACT

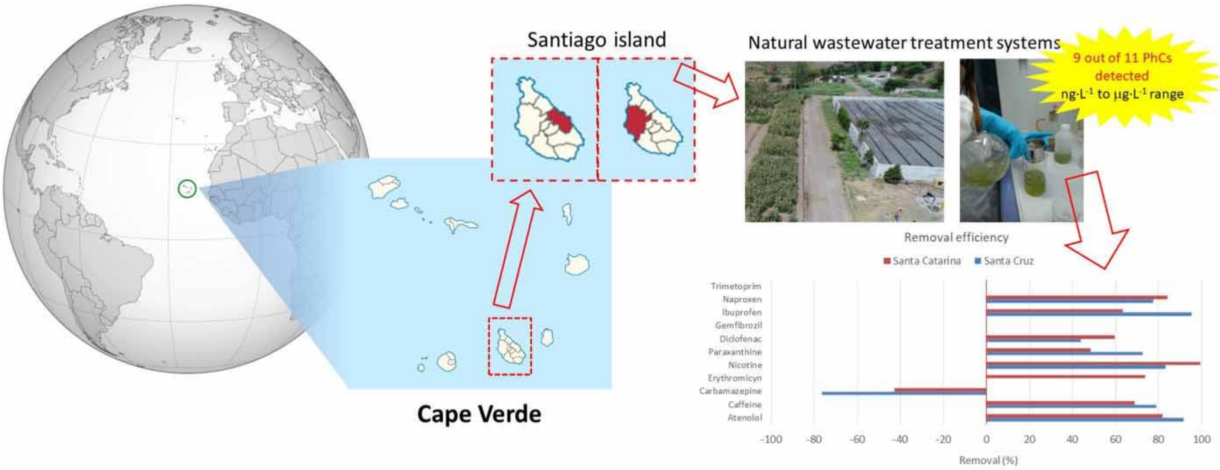
Cape Verde, a West Africa Island country, is exposed to climate change effect. An effective way of reducing its poverty and achieving food security is to provide its agricultural sector with water. The purpose of this study is to monitor treated water quality to ensure that any pollutants are below the limits set to ensure the safety and sustainability of reuse because only 10% of its treated wastewater is reused. For the first time on Cape Verde islands, besides chemical quality, the occurrence of 11 pharmaceutical compounds (PhC) in the influent and effluent of two natural wastewater treatment plants (WWTPs) was monitored to assess the effectiveness of the treatment technologies and removal efficiencies of these pollutants. Of the 11 target PhC, nine were detected in influent samples within the ng·L^{-1} to μg·L^{-1} range. Lower concentrations were detected in one WWTP vs. the other. Nevertheless, the removal efficiencies for most compounds were similar in both WWTPs: within the ranges of 82–92% for beta-blockers, 48–99% for stimulants and 44–95% for nonsteroidal anti-inflammatories, while carbamazepine (anticonvulsant) concentrations increased in effluents. WWTPs produce water that meets water quality standards for irrigation if a properly designed and managed reusing system is adopted.

Key words: emerging pollutants, food safety, pharmaceutical residues, removal efficiencies, treated water quality, wastewater treatment plant

HIGHLIGHTS

- Pharmaceuticals in influent/effluent from two WWTPs were first studied in Cape Verde.
- Nine pharmaceuticals were detected in influent samples of WWTP (ng·L^{-1}–μ·L^{-1} range).
- Erythromycin was detected in the WWTP which receives hospital sewage.
- Achieved removals are similar to analogous systems located on semi-arid islands.
- Proper water management assures negligible risk for pharmaceutical uptake by plants.

GRAPHICAL ABSTRACT



1. INTRODUCTION

Cape Verde is an island country located in the Atlantic area of Macaronesia that lies in the sub-Saharan African climatic zone. Due to its geographic location and specificity, this small country is extremely exposed to natural disasters. Thus, climate change is expected to increase Cape Verde's exposure to extreme weather events. Recently, Cape Verde has witnessed a drastic reduction in rainfall, which results in water shortage and major crop losses, and severely impacts rural livelihoods. All this implies approximately 30,000 people requiring emergency drought assistance. This country is considered a low-/mid-income country (World Bank 2020). Therefore, this is not a local problem because the number of undernourished people in the world has risen from 604 million in 2014 to 768 million in 2020 (FAO 2021). Agriculture in Cape Verde very much depends on rainfall because it has only 2,780 ha equipped for irrigation using both surface (86%) and subsurface (14%) water resources. It represents 3.52% of the total agricultural area of 79,000 ha (FAO 2022), and this agricultural area is 9% of the total land (INE 2018). Santiago, the biggest island of Cape Verde, uses the largest area for agriculture (52%) (Monteiro *et al.* 2020). Its agricultural sector associated with livestock is the main economic activity of municipalities despite low water availability, and it represents the livelihood of many families, mainly women. Agricultural sector growth is one of the most effective ways for reducing poverty and achieving food security in rural areas (FAO 2022).

One of the responses and actions promoted by Food and Agriculture Organization (FAO) in relation to land and water resources for food and agriculture (FAO 2021) includes caring for neglected soils, addressing drought and coping with water scarcity by adopting new technologies and management approaches. Over time, this situation can be affected by the long drought periods that take place. So given this context, it is necessary to find other alternative sources like reusing treated wastewater for agriculture. As pointed out by Mendoza-Grimón *et al.* (2019), nonconventional resources are one of the alternatives that can alleviate the hydrological imbalance between water consumption and renewable resource availability.

Treated wastewater reuse can be considered a reliable water supply that is quite independent of seasonal drought and weather variability and is able to cover water demand peaks (EU 2020/741). Planned reuse with suitable water quality prevents environmental damage from uncontrolled treated or untreated water discharge.

A recent Cape Verde regulation includes treated water irrigation (Decreto Regulamentar no. 4/2020) to promote the safety and sustainability of reuse. Given the significant improvements made to its water treatment stations, now it is possible to provide treated water in compatible quantity and quality terms with irrigation water use. Therefore, in this context, reclaimed water can significantly contribute to strengthen resilience and to adapt to climate change capabilities by enhancing the adaptive capacity to address additional desertification and land degradation risks (Mendoza-Grimón *et al.* 2021). Yet despite its benefits, reclaimed water reuse currently poses some barriers, such as its environmental and health risks, which are perceived by consumers and limit its exploitation. Cape Verde and the European Union through Interreg-MAC Projects have invested and encouraged the practice of reusing treated wastewater in agriculture. The main objectives are to address the lack of water resources in regions with agricultural potential and to protect the environment, especially in coastal waters, by reducing the pollutant load released to the sea and to groundwater. However, reused treated wastewater is very limited and does not exceed 10% of the total nationally treated wastewater (ANAS 2018).

Reusing treated water in agriculture is essential in water-scarce areas to guarantee economic and environmental sustainability in semi-arid regions (Palacios-Díaz *et al.* 2015). In addition, treated water is the only resource that grows with increasing needs. However, it is necessary to evaluate and monitor this treated water to ensure that any pollutants present, including emerging products like antibiotics, analgesics and antiseptics, are below the limits that pose dangerous health and environmental effects. These compounds, which represent one of the most important pharmaceutical groups in Cape Verde, have different applications in human and veterinary medicine, such as growth promoters in livestock (Lopes *et al.* 2022). Many of these applications are performed with no advice from a doctor or veterinarian and, thus, endanger their own animal and food safety. Despite the positive effects of antibiotics, using these drugs without medical advice can give rise to increased antibiotic resistance to pathogenic bacteria, whose effects can pose a potential threat to ecosystems and human health (Christian *et al.* 2003; Kümmerer 2009).

Recently, several studies have detected pharmaceutical residues in different water samples, such as municipal wastewater, groundwater, river water and even drinking water (Ofrydopoulou *et al.* 2002; Fick *et al.* 2009; ANSES 2011; Estevez *et al.* 2016; Samaraweera *et al.* 2019; Golovko *et al.* 2021), and also on semi-arid Macaronesian islands like the Canary Islands (Guedes-Alonso *et al.* 2020). These polluting compounds are constantly introduced into the environment because the treatment processes used by effluent treatment plants are not yet effective enough to completely remove them. Their introduction into the environment can be *via* point or diffuse sources through the application of residues, the addition of manure of animal origin to soil or the irrigation of agricultural soils with treated wastewater (Abdallah *et al.* 2019; Golovko *et al.* 2021). Recently, more attention to emerging contaminants (Ecs) in the environment has been paid, particularly in wastewater, given their adverse effects on the aquatic environment (Cabello 2006; Krakkó *et al.* 2019) and soils (Kümmerer 2004; Vazquez-Roig *et al.* 2012). Studies have highlighted not only the toxic effects of drug residues on algae and crustaceans but also the reduction of certain marine species (Stoecker *et al.* 2006; Teixeira & Granek 2017).

It is also important to monitor the wastewater that enters treatment plants to protect public health because wastewater monitoring can provide information about the pattern of drug use in a population of a served area. One of the three main objectives of the ADAPTaRES Project (the EU Interreg-MAC project which financed this study) is to guarantee the quality of treated wastewater for agricultural use in Macaronesia. Focusing on Cape Verde, this project carried out an unprecedented study to evaluate and quantify pharmaceutical residues in influent and effluent wastewaters because, even now, there are no studies that report the concentration of these residues in water for this country.

Therefore, the main purpose of this study is to disseminate the results obtained from a 1-year Ecs assessment (11 pharmaceutical compounds, including antibiotics), in both the influent and effluent, to know the effectiveness of the treatment technologies and removal efficiencies (REs) of these pollutants. This information is needed to create a database of these pollutants to assist the Cape Verde government to regulate these pollutants in water samples by promoting safety water reuse in agriculture. With the provided information, the government will be able to take measures to combat antimicrobial resistance in the environment based on the World Health Organization's global plan of action.

2. METHODS

2.1. Reagents and solvents

In this study, Atenolol, Caffeine, Carbamazepine, Erythromycin, Nicotine, Paraxanthin, Diclofenac, Gemfibrozil, Ibuprofen, Naproxen and Trimethoprim (all from Sigma-Aldrich, Madrid, Spain) were studied in the chosen wastewater treatment plants (WWTPs). The selection of analytes was based on population surveys in the study areas and at the local pharmacies where prescription and nonprescription drugs were purchased. The purities of the target pharmaceuticals were over 97%. Three internal standards (IS) atenolol-d7, ibuprofen-d3 and sulfamethoxazole-d4 (Toronto Research Chemical Inc., Toronto, Canada, Sigma-Aldrich, Madrid, Spain and Dr Ehrenstorfer GmbH, Ausgburg, Germany, respectively) were used for the determination of the target analytes. Individual stock solutions were prepared in methanol at a concentration of 1,000 $mg{\cdot}L^{-1}$ and were stored in the dark and in a freezer at –20 °C. A working mixture of these stocks was also prepared in methanol at a concentration of 10 $mg{\cdot}L^{-1}$ and stored under the same conditions as individual standards. LC-MS methanol and LC-MS water, both with 0.5% acetic acid, were employed as the chromatographic mobile phase. All these solvents were supplied by Panreac (Barcelona, Spain). The ultrapure water used in the sample preparation procedure (Milli-Q water) was obtained by a water purification system (Millipore, Bedford, MA, USA).

2.2. Sampling and study area

Two municipal WWTPs located on the Santiago Island (the Santa Cruz and Santa Catarina WWTPs) were monitored in this study (Table 1). The maximum designed flow rate of the Santa Cruz WWTP is 1,000 $m^3 \cdot day^{-1}$, with a maximum of 225 $m^3 \cdot day^{-1}$ for the Santa Catarina WWTP. They currently treat 200 and 144 $m^3 \cdot day^{-1}$, respectively (INE 2018). The major difference between the two WWTPs lies in the treatment process: both WWTPs employ pretreatment with flotation, desander/degreaser and screening. The Santa Catarina WWTP has a primary treatment pond, followed by a discontinued discharged system to a secondary vertical filtration system. The Santa Cruz WWTP has a primary and secondary decanter system with anaerobic digestion, followed by a secondary infiltration treatment (ANAS 2016; ADAPTARES 2020). The Santa Cruz WWTP treatment process is carried out naturally with no chemical agent or energy use. Finally, the treated effluents from both plants are stored for later reuse in agriculture. The Santa Catarina storage pond allows a disinfection process by receiving ultraviolet rays.

Sampling was carried out in both WWTPs during two periods (May 2018 and December 2020) to determine water quality characterization, including the REs of Ecs. In 2018, samples were taken at the inlet and outlet in triplicate. In the Santa Catarina WWTP, samples were taken at an additional sampling point after a filtration system in 2020, and also in triplicate. This system was installed to prevent clogging problems in a drip irrigation system employed to reuse water. All the samples were collected as a grab sample using 1-liter glass bottles. Before sampling, glass bottles were washed with the samples on three consecutive occasions. Samples were immediately transported to the Public University of Cape Verde (Uni-CV) laboratory, protected from light and refrigerated at 4 °C in a thermostatic box. Water chemical characterization was performed in INLAB, a local laboratory, or in the facilities of the Laboratorio Fitopatológico y Agroalimentario del Cabildo de Gran Canaria (LAFGC, Spain), for which they were frozen and transported under conditions that did not affect the analysis parameters. For the pharmaceutical analysis, sample pretreatment was carried out at the Uni-CV laboratories, and the determination of the target analytes was performed in collaboration with the Environmental Chemical Analysis Group of the University of Las Palmas Gran Canaria.

2.3. Water quality characterization

The treated water characteristics were analyzed by INLAB and used in the internal method based on Decree Law 236 of 1,998 of Portugal. The water characterization at LAFGC was performed by the following methods: pH and Electrical Conductivity (EC) were analyzed by an electrometric method. Water samples were filtered through a 0.45 μm-pore membrane filter. Chemical oxygen demand (COD) was analyzed with a test kit (reference 0–29, 1,500, Nanocolor CSB), biochemical oxygen demand after 5 days (BOD_5), by measuring oxygen and total suspended solids (TSS), respectively, by selective electrode and APHA (1992). For the metal and metalloid quantifications, determined by atomic emission spectroscopy optical emission (ICP-OES analysis), a filtered aliquot sample was acid-stabilized ($pH < 2$) and stored at 4 °C until the time the analysis was done. A second aliquot was stored as frozen until the quantification for the major anions (Cl^-, NO_3^- and SO_4^{2-}), which were determined by an ion chromatography analysis. Total nitrogen (N_{tot}) was determined following the Kjeldahl

Table 1 | Natural wastewater treatment plants (WWTPs) monitored in this study

	Santa Cruz WWTP	Santa Catarina WWTP
Max designed flow rate ($m^3 \cdot day^{-1}$)	1,000	225
Current flow rate ($m^3 \cdot day^{-1}$)	200	144
Pretreatment	Flotation, desander/degreaser and screening	Flotation, desander/degreaser and screening
Primary treatment	Decanter system with anaerobic digestion	Pond
Discharged system to secondary		Discontinued
Secondary treatment	Decanter system with anaerobic digestion Infiltration treatment	Vertical filtration
Storage		Pond with disinfection process by UV

methodology. The sodium adsorption ratio (SAR) was calculated by the following equation:

$$\mathrm{SAR} = \frac{[\mathrm{Na}]}{\sqrt{\left(\frac{[\mathrm{Ca}] + [\mathrm{Mg}]}{2}\right)}} \quad (1)$$

2.4. Pharmaceutical analysis

Before extracting compounds, 750 mL of the influent and effluent samples were adjusted to a pH of 7 ($\pm$0.1) and filtered through 1 μm glass fiber filters to eliminate any suspended solids that may lead to the clogging of the solid phase extraction (SPE) cartridges used to extract the target analytes. Cartridges were placed inside an SPE extraction device fitted with a Supelco Visiprep™ vacuum manifold. They were preconditioned using 5 mL of methanol and 5 mL of ultrapure water. A 250 mL aliquot of the wastewater samples (in triplicate) was passed through cartridges at a constant flow rate of 1 mL·min^{-1}. When sample loading ended, cartridges were dried for 20 min in a vacuum and sent safely to the University of Las Palmas Gran Canaria for the pharmaceutical determination. The stability of the sorbed pharmaceuticals in the SPE cartridges has been previously studied and reported by Carlson *et al.* (2013), who demonstrated that pharmaceuticals are stably adsorbed in SPE cartridges for a long period. This procedure permits the target compounds to be extracted in a short time after sampling campaigns to avoid compound degradation.

To perform the pharmaceutical compounds analysis, cartridges were washed with 5 mL of ultrapure water (Milli-Q water) and the retained compounds were eluted with 5 mL of methanol. To achieve adequate preconcentration factors, extracts were evaporated in nitrogen and reconstituted with 1 mL of Milli-Q water with 100 μg·L^{-1} of IS. Before the analysis, extracts were filtered using Chromafil Xtra PET-20/25 syringe filters (a pore size of 0.20 μm; Machery-Nagel, Düren, Germany).

Extracts were analyzed by ultrahigh-performance liquid chromatography tandem mass spectrometry (UHPLC-MS/MS). To perform the separation of the target analytes, an ACQUITY UPLC BEH Waters C18 column (50 mm $\times$ 2.1 mm, 1.7 μm) from Waters Chromatography (Barcelona, Spain) was used. The mobile phase consisted of water (A) and methanol (B), both with 0.5% acetic acid. The determination conditions were optimized in a previous study (Guedes-Alonso *et al.* 2020). Briefly, detection was performed by electrospray ionization in both the positive (ESI+) and negative (ESI–) modes.

Regarding the analytical parameters, the method was evaluated for linearity, sensitivity, reproducibility and extraction efficiency. To perform the calibration of the method, the addition of IS (atenolol-d7; Toronto Research Chemical Inc, Toronto, Canada, ibuprofen-d3; Sigma-Aldrich, Madrid, Spain and sulfamethoxazole-d4; Dr Ehrenstorfer GmbH, Ausgburg, Germany) was used so that the matrix effect produced by the interferences extracted during SPE was minimized. In this way, the linearity of the method was evaluated by comparing the analytical signals of each compound to the corresponding IS within a range from 1 to 400 μg·L^{-1}. The concentration of the IS was left constant at all the calibration curve points. For all the compounds, very satisfactory linearity was observed with regression coefficients (r^2) higher than 0.990. The instrumental limits of detection and quantification (LOD and LOQ, respectively) were calculated as the concentration that provides a signal-to-noise ratio equaling 3 and 10, respectively. For all the target analytes, the LODs and LOQs were appropriate for the environmental analysis. LODs were between 16 and 671 ng·L^{-1} (except naproxen, with a LOD = 7,712 ng·L^{-1}). For many compounds, such as atenolol, caffeine, carbamazepine, erythromycin, nicotine, paraxanthine and trimethoprim, LODs were below 180 ng·L^{-1}. Regarding the repeatability of the analysis method, the relative standard deviations (RSDs) for each compound were evaluated at a low concentration level (200 ng·L^{-1}) both intra- and interday. The RSDs for the target analytes in intraday repeatability were between 5.6 and 14.4%, while the interday RSDs fell within the range from 9.4 to 18.1% (Afonso-Olivares *et al.* 2017). Finally, the recoveries of the extraction procedure were also previously optimized and were very satisfactory for all the analyzed compounds. The extraction efficiencies of the SPE procedure at a low concentration level (200 ng·L^{-1}) fell within the range from 92 to 116.1% in the effluent samples and between 79.4 and 125.1% in the influent samples (Afonso-Olivares *et al.* 2017).

2.5. Removal efficiencies

Removal efficiency (RE) expresses the elimination of a compound as a percentage during treatment. It is calculated by the ratio of the difference between the influent and effluent wastewater during the sampling period with the following equation:

$$\mathrm{RE}\ (\%) = \frac{C_{\mathrm{inf}} - C_{\mathrm{eff}}}{C_{\mathrm{inf}}} \cdot 100 \quad (2)$$

where C_{inf} refers to the concentrations of the Ecs detected in the influent, and C_{eff} denotes the concentrations of the Ecs detected in the effluent, and both in each WWTP.

3. RESULTS AND DISCUSSION:

3.1. Water quality characterization

The Cape Verde Regulation (Decreto Regulamentar no. 4/2020) establishes the criteria and parameters for controlling the quality of: water for irrigation; surface or groundwater; desalinated, recovered rainwater or treated wastewater. The maximum admissible value (VMA) and the maximum value recommended (VMR) are established for some parameters. Their normative-based restriction levels for other parameters follow the Ayers & Westcot (1985) recommendations for the degree of restrictions about agricultural irrigation water use.

As Table 2 shows, the water quality produced by both WWTPs remained stable over time. The Santa Catarina WWTP obtained high values for COD, BOD5, SAR, EC, Cl, Na, nitrate, N_{tot}, P_{tot} and TSS. The Santa Cruz WWTP only produced high values for EC, Cl, Na, nitrate and sulfate.

Regarding the pH of the water effluents, which fell within the VMR and VMA ranges, no irrigation restrictions must be imposed. ECs were higher than VMR and at the upper limits of VMA and presented a severe restriction for irrigation use from both WWTPs. However, the marked presence of salts other than conventional ones can modify the salinity threshold levels tolerated by crops. COD and BOD_5 are not included in the Cape Verde Regulation but, when considering Regulation (EU) 2020/741 on minimum requirements for water reuse, the Santa Cruz water can be considered to be A quality class. The Santa Catarina water values were higher than any quality class, which expects additional treatment before irrigation and careful water management. According to the Cape Verde Regulation, TSS was below and higher than VMA in the Santa Cruz water and the Santa Catarina water, respectively, while Ca, Mg and sulfate were below VMR for both WWTPs. B, Fe, Mn

Table 2 | Means and standard deviations of the water chemical parameters from the Santa Catarina and Santa Cruz water effluents

Parameters (units)	Santa Catarina	Santa Cruz
pH	7.90 ± 0.29	7.35 ± 0.21
EC ($dS·m^{-1}$)	3.25 ± 0.26	3.09 ± 0.16
COD ($mg·L^{-1}$)	212	32
BOD_5 ($mg·L^{-1}$)	60	6.3
TSS ($mg·L^{-1}$)	154.7	2.1 ± 0.04
HCO_3^- ($mg·L^{-1}$)	530 ± 10	n.a.
Na^+ ($mg·L^{-1}$)	438 ± 3	340
K^+ ($mg·L^{-1}$)	95.3 ± 1.7	83.0
Ca^{2+} ($mg·L^{-1}$)	64.3 ± 5.9	93.2 ± 2.6
Mg^{2+} ($mg·L^{-1}$)	45.3 ± 1.3	75.2 ± 5.4
Cl^- ($mg·L^{-1}$)	573.3 ± 24.9	427.5 ± 17.7
N_{tot} ($mg·L^{-1}$)	130	n.a.
NO_3^- ($mg·L^{-1}$)	250.7 ± 27.8	410.0 ± 127.3
P_{tot} ($mg·L^{-1}$)	37	n.a.
SO_4^{2-} ($mg·L^{-1}$)	39.5 ± 3.5	110
SAR $(meq·L^{-1})^{1/2}$	10.23 ± 0.26	6.51 ± 0.41
B ($mg·L^{-1}$)	0.165 ± 0.005	n.a.
Cu ($mg·L^{-1}$)	< 0.015	n.a.
Fe ($mg·L^{-1}$)	0.077	n.a.
Zn ($mg·L^{-1}$)	0.011	n.a.
Mn ($mg·L^{-1}$)	0.1755 ± 0.1255	n.a.

n.a., not analyzed.

and zinc were only determined in the reclaimed water from Santa Catarina WWTP plant and remained below VMR. Finally, Na, Cl and nitrate presented a severe restriction for irrigation use (and, thus, require careful management), while SAR and B posed no irrigation restriction for both WWTPs. For the above nitrate limits, and considering the marked N extractions of the C4 crops (Palacios-Díaz *et al.* 2013) that are frequently grown in this country, the N level considered by the Cape Verde Regulation must be revised. Higher levels can be permitted in treated water because high plant absorption often exceeds the N supplied by reclaimed water when considering the doses used for irrigation. Therefore, natural WWTPs from rural communities produce water that meets water quality standards and avoids groundwater contamination at the same time.

3.2. Occurrence and concentrations of the target pharmaceuticals

Table 3 summarizes the values in terms of the concentration of the pollutants studied at the two stations selected for this case study. Of the 11 target pharmaceuticals, nine were detected in influent samples in either WWTP. No gemfibrozil and trimethoprim concentrations were detected in any sample during this study. There were some differences between the concentrations detected in both surveyed WWTPs. For example, erythromycin was not detected in the Santa Cruz WWTP, but it was the compound with the highest concentration in the Santa Catarina influent wastewater (31 $\mu g \cdot L^{-1}$). A difference was observed for two of the studied nonsteroidal anti-inflammatory drugs (NSAIDs), ibuprofen and naproxen, which were, respectively, 2.7- and 4-fold higher in the Santa Catarina WWTP influent. For these two compounds, the concentrations recorded in both WWTPs were between 1.3 and 7.8 $\mu g \cdot L^{-1}$, which are lower than those reported in similar treatment systems on Gran Canaria, another island in the Macaronesia region (Guedes-Alonso *et al.* 2020, 2022). Santa Catarina, with a 55% bigger population than Santa Cruz (INE 2020), has a WWTP that effectively treats 64% of its designed capacity, while Santa Cruz treats only 20% of its capacity. This means that the Santa Catarina WWTP treats 3 L per habitant, while the Santa Cruz WWTP treats 7 L per habitant. This fact can partially explain the lower concentrations of the pharmaceutical compounds detected for Santa Cruz than for Santa Catarina. This result can also be explained by the effluents received by the Santa Catarina WWTP from a regional hospital (Santiago Norte), which are mixed with the region's domestic effluents. Thus before pouring hospital effluents, it is necessary to subject them to an advanced treatment. Another major difference between the surveyed WWTPs was the nicotine concentration. In the Santa Catarina WWTP, the influent concentration was 16-fold higher than in the Santa Cruz WWTP influent, with an average concentration of 5.6 $\mu g \cdot L^{-1}$. The nicotine concentrations detected for Santa Catarina were similar to those obtained in a rural WWTP of Gran Canaria (Canary Islands). The average nicotine concentrations detected in the Santa Cruz WWTP influent (350 $ng \cdot L^{-1}$) fell within a lower range than those reported for similar WWTPs of Macaronesia (Guedes-Alonso *et al.* 2020, 2022).

After the treatment system had ended, the nicotine concentrations in both WWTPs were similar (31.6–58.6 $ng \cdot L^{-1}$). The influent concentrations for another studied NSAID, diclofenac, were low (between 0.15 and 0.4 $\mu g \cdot L^{-1}$) and fell within the

Table 3 | Means of the water chemical parameters (expressed as $ng \cdot L^{-1}$) from the Santa Catarina and Santa Cruz water effluents

	Santa Cruz WWTP		Santa Catarina WWTP	
Compounds ($ng \cdot L^{-1}$)	**Influent**	**Effluent**	**Influent**	**Effluent**
Atenolol	349.2	28.8	454.5	82.0
Caffeine	1,692.3	357.8	1,006.5	311.7
Carbamazepine	1,054.2	1,859.2	391.2	558.3
Erythromycin	n.d.	n.d.	31,020.4	8,154.0
Nicotine	350.3	58.6	5,611.5	31.6
Paraxanthine	813.4	222.3	259.9	133.9
Diclofenac	156.1	87.6	399.5	160.5
Gemfibrozil	n.d.	n.d.	n.d.	n.d.
Ibuprofen	2,863.7	134.4	7,819.7	2,851.5
Naproxen	1,285.9	290.3	5,127.3	819.0
Trimetoprim	n.d.	n.d.	n.d.	n.d.

n.d., not detected.

same range as other wastewater treatment facilities. No differences were observed in the concentrations of caffeine and its main metabolite, paraxanthine, at both treatment facilities, where caffeine concentrations were higher than paraxanthine. This has also been observed in a macrophyte pond-constructed wetland system studied on Gran Canaria (Guedes-Alonso *et al.* 2022). In both the Cape Verdean WWTPs, the influent concentrations of these two stimulants (up to 1.6 $\mu g \cdot L^{-1}$) were very low compared to other studies that report caffeine concentrations in influent samples exceeding 30 $\mu g \cdot L^{-1}$ (Guedes-Alonso *et al.* 2020, 2022). Finally, the recalcitrant behavior of carbamazepine was also observed in both surveyed WWTPs. Its average concentration for Santa Catalina was 391.2 $ng \cdot L^{-1}$, but this value rose to 1,054.2 $ng \cdot L^{-1}$ for Santa Cruz. At both facilities, concentrations were higher after the treatment process. This is in accordance with many other studies that highlight the impossibility of eliminating this pharmaceutical by traditional wastewater treatments due to the poor biodegradability and hydrolysis of carbamazepine from a glucuronide conjugate (Kasprzyk-Hordern *et al.* 2009; de Oliveira *et al.* 2020).

3.3. Pharmaceutical removal efficiency

After assessing the concentrations of the target pharmaceuticals in both WWTPs, the removals of each wastewater treatment were calculated. As seen in Figure 1, the REs for most compounds were similar in both WWTPs. Removals were good for the target stimulants. However, different results were obtained at both stations. On the one hand, the removal of caffeine and the main metabolite of caffeine, namely paraxanthine, were higher for Santa Cruz, and were more than 20% higher for Santa Cruz than Santa Catarina for paraxanthine. In contrast, the nicotine removal in the Santa Catarina WWTP was higher (99 vs. 83%). Concerning NSAIDs, better ibuprofen removal was observed for Santa Cruz (95%) compared to Santa Catarina (63%). The differences for the other studied NSAIDs (diclofenac and naproxen) were not as significant, with removals ranging from 44 to 60% for diclofenac, and from 77 to 84% for naproxen. For atenolol, removals were high (above 82%) in both WWTPs. The recalcitrant behavior of carbamazepine was observed in both WWTPs, and its concentrations rose between 43 and 76%, which indicates the low degradation of this pharmaceutical, an aspect that has been widely reported in the literature (Zhang *et al.* 2008; Durán-Álvarez *et al.* 2015). Nevertheless, Durán-Álvarez *et al.* (2015) determined that carbamazepine photodegraded more than it biodegraded, which could explain why the rise in carbamazepine concentrations was not so marked in the Santa Catarina WWTP because it uses a UV disinfection process after the secondary treatment.

Compared to other studies conducted in constructed wetlands (CWs), the removals obtained at the two studied treatment facilities were similar (Table 4). In fact, the removals recorded for atenolol or some stimulants like nicotine or NSAIDs like ibuprofen fell within the same range as a study performed on Gran Canaria, another Macaronesian Island (Guedes-Alonso *et al.* 2020), which evaluated removals in a WWTP based on vertical and horizontal flow CWs. The removals of the other NSAIDs (diclofenac and naproxen) were better than the results obtained on the Gran Canaria Island (Guedes-Alonso

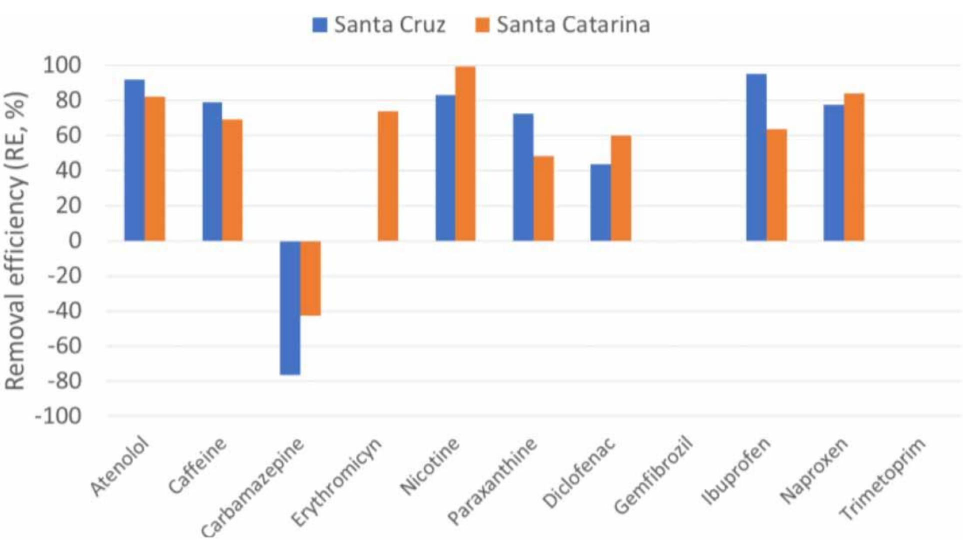


Figure 1 | Removal efficiencies (REs) of the detected pharmaceuticals at the Santa Cruz and Santa Catarina WWTP.

Table 4 | Comparison of pharmaceutical removals in the WWTPs based on constructed wetlands

Therapeutic family	Compound	Removal (%)	Reference
Beta-blockers	Atenolol	>90	Guedes-Alonso *et al.* (2020)
		85	Vymazal *et al.* (2017)
		82–92	**This study**
Stimulants	Caffeine	>97	Guedes-Alonso *et al.* (2020)
		>95	He *et al.* (2018)
		>85	Hijosa-Valsero *et al.* (2016)
		80–90	Vystavna *et al.* (2017)
		69–79	**This study**
	Paraxanthine	>97	Guedes-Alonso *et al.* (2020)
		48–73	**This study**
	Nicotine	>97	Guedes-Alonso *et al.* (2020)
		83–99	**This study**
Nonsteroidal anti-inflammatories	Diclofenac	<20	Hijosa-Valsero *et al.* (2016)
		5–95	He *et al.* (2018)
		40	Vymazal *et al.* (2017)
		60–95	Vystavna *et al.* (2017)
		44–60	**This study**
	Ibuprofen	79	Guedes-Alonso *et al.* (2020)
		20–80	He *et al.* (2018)
		22–78	Hijosa-Valsero *et al.* (2016)
		55	Vymazal *et al.* (2017)
		55–60	Vystavna *et al.* (2017)
		64–95	**This study**
	Naproxen	36	Guedes-Alonso *et al.* (2020)
		40–90	He *et al.* (2018)
		60–80	Hijosa-Valsero *et al.* (2016)
		85	Vystavna *et al.* (2017)
		77–84	**This study**
Anticonvulsant	Carbamazepine	−50	Guedes-Alonso *et al.* (2020)
		3–47	He *et al.* (2018)
		20	Hijosa-Valsero *et al.* (2016)
		42–76	**This study**

Note: Bold values are the results obtained by this authors in this work.

et al. 2020), but were similar to other studies that have evaluated the removal of these compounds in CWs systems (Hijosa-Valsero *et al.* 2016; Vymazal *et al.* 2017; Vystavna *et al.* 2017). The carbamazepine concentrations rose after treatment and fell within a range like that reported in the study conducted on the Gran Canaria Island (Guedes-Alonso *et al.* 2020). Other studies have stated that carbamazepine removal can be performed in systems with a high biomass (Hijosa-Valsero *et al.* 2016). Nonetheless, both studied WWTPs were based on unplanted CWs, which could explain the obtained negative removal ratios.

In a previous study (Mendoza-Grimón *et al.* 2022), which artificially added pharmaceuticals at high levels (100 $\mu g \cdot L^{-1}$, about 100-fold higher than those detected in this experiment) to irrigation water, no pharmaceutical over LODs (0.06–0.6 $\mu g \cdot kg^{-1}$of dry plant sample) were detected in the plant samples. This finding indicates the importance of irrigation systems, water management and the soil–plant barrier to avoid pharmaceuticals uptake. Therefore, given the values detected in this experiment, a negligible risk for pharmaceuticals uptake can be considered if proper irrigation design and water management are used.

4. CONCLUSIONS

WWTPs produce water that meets water quality standards for irrigation if a properly designed and managed reusing system is adopted. The water quality obtained by the studied Cape Verde WWTPs remained stable over time. The Santa Catarina WWTP produced high values for COD, BOD5, SAR, EC, Cl, Na, nitrate, N_{tot}, P_{tot} and TSS (requiring additional filtration treatment for drip irrigation), while the Santa Cruz WWTP obtained high values only for EC, Cl, Na, nitrate and sulfate.

Of the 11 target PhC, nine were detected in influent samples within the ng·L^{-1} to μg·L^{-1} range of both WWTPs, but differences appeared between the detected concentrations. For example, erythromycin was detected only for Santa Catarina at 31 and 8 μg·L^{-1} in influent and effluent, respectively. Lower concentrations were detected in the Santa Cruz WWTP vs. the Santa Catarina WWTP, which would probably be affected by hospital sewage entering the Santa Catarina WWTP, and the REs for most compounds were similar in both WWTPs: fell within the ranges of 82–92% for beta-blockers, 48–99% for stimulants and 44–95% for nonsteroidal anti-inflammatories, while carbamazepine concentrations increased in effluents. The REs for most compounds were similar in both WWTPs. Per group, they fell within the following ranges: 82–92% for atenolol, 48–99% for stimulants and 44–95% for NSAIDs. The carbamazepine concentration increased in effluents and displayed recalcitrant behavior according to studies conducted in other wastewater treatment systems of the Macaronesia region. However, more studies are necessary to understand the evolution of pharmaceutical compounds in the water–soil and plant systems affected by water management, even by considering the low detected values obtained in our experiment to guarantee environmental protection. Besides, a negligible risk for pharmaceuticals uptake can be considered if proper irrigation design and water schedule are used.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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