



Research article

Impact of a WWTP effluent overland flow on the properties of a mediterranean riparian soil

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ABSTRACT

In this work we aim to assess the impact of a WWTP effluent overland flow on properties and nutrient concentrations of a riparian soil, in order to explore the potential of this practice as a nature-based treatment. We set two study zones of 150 m² on the field, one control and one that received the WWTP effluent on its surface for one month. Samples were taken before and after the effluent overland flow system, to test the impact of the effluent on soil properties through a BACI design, and after 17 months, to evaluate the recovery of the soil. Two depths were studied: 0–5 cm and 5–20 cm. The effluent overland flow triggered an increase in exchangeable sodium percentage and a decrease in nitrate concentration in both depths, and an increase in ammonium concentration in 0–5 cm depth. After 17 months, there were not found relevant differences among zones. In conclusion, this practice could be used in the purpose to reduce the nutrient concentrations of WWTP effluents. This practice could be relevant for regions where WWTP effluents are discharged in low-flow or intermittent streams, such as semi-arid regions or the Mediterranean region.

1. Introduction

Riparian zones have a great water purification capacity (Vought et al., 1994; Mander et al., 2005), and they play an important role in the removal and retention of nutrients such as nitrogen and phosphorus (Vidon et al., 2010; de Sosa et al., 2018). This capacity has already been employed as a management tool to improve water quality from diffuse sources in riparian buffer zones (RBZ) (Muscott et al., 1993; Mosquera-Losada et al., 2018). Therefore, they have the potential to reduce and transform nutrients from wastewater treatment plant (WWTP) effluents. In the last decades, wastewater tertiary treatments have been carried out in-stream and near stream to improve the quality of WWTP effluents and reduce their impact on freshwater ecosystems. Examples include constructed wetlands (CW) in the flooded land (Zheng et al., 2014), vegetated buffer zones (Laurent et al., 2015; Koenig and Trémolières, 2018), and the discharge of WWTP effluents over floodplains (EPA, 2018; Batista Seguí et al., 2019) and in conveyance channels (Narr et al., 2019) or overland flow systems.

The main processes that take place in RBZ to reduce pollutants are physical retention, plant uptake, dilution, and chemical transformation (Osborne and Kovacic, 1993). In fact, the main path of nitrate removal is via denitrification thanks to the abundance of both water and organic matter in soils (Sabater et al., 2003; Vidon et al., 2010), and the occurrence of hot spots and hot moments that trigger higher denitrification rates (McClain et al., 2003). Regarding phosphorus, riparian zones have a great potential to intercept total P in overland flow (Väänänen et al., 2006) and to adsorb P onto soil particles (Lyons et al., 1998), although redox conditions may dictate whether they act as a sink or a source of P (Vidon et al., 2010). For available phosphorus, it is also difficult to predict the retention capacity because of the complex interactions between hydrological and biogeochemical processes that condition if the RBZ (Martin and Reddy, 1997; Walton et al., 2020). Nevertheless, it is reported that long-term application of WWTP effluents changes soil properties, resulting in an increase of pH and organic matter content (Walker and Lin, 2008). Of special relevance is the impact that effluents may have on soils regarding salt accumulation,

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sodicity and, therefore, soil structure (Lado and Ben-Hur, 2009). On the other hand, in arid and semiarid zones it has been reported that 500 mm average precipitation is enough to leach salts below the root zone and prevent long-term salt accumulation in soils irrigated with secondary effluents (Lado and Ben-Hur, 2009). Furthermore, some studies have reported small changes in nutrient concentrations in soils during short-term effluent applications, such as a decrease in nitrate and increase in ammonium in soils (Duan et al., 2010; Tzanakakis et al., 2011; Barbagli et al., 2019). However, the potential of riparian forest soils as a nature-based alternative tertiary treatment of WWTP effluents through an overland flow (OF) on field scale has not been sufficiently studied, especially in Mediterranean regions. In this sense, this is a novel approach to consider since there is no available information concerning this practice.

This study examined the impacts of a WWTP effluent overland flow on the riparian soil of a small, intermittent Mediterranean stream in Catalonia. Our objective was to assess the short-term impact of a WWTP effluent overland flow on the properties and nutrients content of a riparian soil, both in depths 0–5 cm and 5–20 cm, and to evaluate the recovery after 17 months. Given the context above, we hypothesized that the effluent overland flow would facilitate the reduction of nitrate concentration in soil because of the occurrence of anaerobic conditions. On the other hand, we would expect an increase in ammonium, especially in topsoil since the effluent is rich in this compound. As we were evaluating the effects of a short-term application, we did not expect significant changes in either dissolved organic carbon, phosphorus or in parameters related to salt content and pH. We expect our study to contribute to assess the capacity of riparian forest soils to process and transform the nutrients from WWTP effluents in field conditions, in which case it might become a nature-based alternative tertiary treatment. Besides, this practice could decrease the impact that WWTP effluents have on intermittent and low-flow streams in some Mediterranean and semi-arid regions (Hassan and Egozi, 2001; David et al., 2013; Bernal et al., 2020). There are several studies about constructed wetlands or alternative tertiary treatments developed on riparian zones in Mediterranean regions (Gunes et al., 2012; Morugán-Coronado et al., 2011; Tzanakakis et al., 2011; Tuncsiper, 2018; Lavrnić et al., 2020). Nonetheless, we have not found nature-based solutions applied in a field scale, in which there were not constructed structures involved without artificial substrate (sediments or soil), limited space by walls or channelized ditches, and/or plantations. On the other hand, there are studies where riparian meadows were irrigated with WWTP effluents in temperate regions (Batista Seguí et al., 2019), but they differ from our study case concerning the type of riparian ecosystem and climate region. For these reasons, our experiment constitutes a novel attempt to determine the potential of a riparian forest soil in field conditions to treat WWTP effluents in a Mediterranean region. Additionally, we see it as a good contribution to better understand the performance of these systems at the field scale, which has been neglected in studies of wastewater treatment in constructed wetlands in the last years (Vymazal et al., 2021).

2. Materials and methods

2.1. Study site

The study was carried out at the municipality of Cànoves i Samalús in the mid-northeast of Catalonia (41° 41' 39.63" N, 2° 21' 12.02" E). The climate is Mediterranean with wet springs, warm and dry summers, and mild winters. The average yearly precipitation is between 700 mm and 1000 mm. During the study period, annual precipitation was 550 mm in 2019 and 873.5 mm in 2020 (Catalan Meteorological Service). The relief is quite mountainous due to its close location to Montseny massif and tertiary materials like clay, sandstones and conglomerates from the Miocene are found in the zone (Catalan Cartographic Institute). Forests cover 77% of the surface, while agricultural land use represents 15%.

Urban land cover represents only 5% of the surface, but the urbanized areas are scattered across the municipality.

The specific study site is in the riparian forest next to the wastewater treatment plant (WWTP) of Cànoves i Samalús (Fig. 1a). The WWTP was built in 2006 and it treats the water of 9200 inhabitant equivalents, with a daily mean flow of 104 m³/h. The operation of the plant consists of a pre-treatment and a biological activated sludge treatment. The effluent is discharged to the stream of Cànoves, which has a natural strong seasonal character upstream of the WWTP. During the dry season (summer) it has a flow at or close to 0 m³/s, thus a dilution factor of zero, reflecting the high impact of the WWTP effluent on the stream (Pascual-Benito et al., 2020). Furthermore, the effluent discharge has been reported to be an input of five fecal indicator organisms to the stream of Cànoves, such as *Escherichia coli* and spores of sulphite-reducing clostridia (Pascual-Benito et al., 2020). The riparian forest consists in a riparian vegetation community dominated by *Alnus glutinosa*, with presence of *Platanus x hispanica* Mill., *Populus nigra* and *Fraxinus excelsior*, bushes such as *Sambucus nigra*, and a diversity of riparian herbaceous species such as *Arum italica* and *Carex pendula*.

2.2. Experimental design and operation

The experiment followed a BACI design (Green, 1979). It consisted of two sampling campaigns (before and after) and two 150 m² study zones (control and impact). In 2017, there was a construction work on the WWTP of Cànoves i Samalús that consisted in the installation of a shunt pipe for the WWTP effluent. That shunt pipe was connected to a system of pipes with two discharge points on the riparian forest which allowed the performance of the effluent overland flow through the surface of the impact zone (herein, treated zone) (Fig. 1b). The effluent overland flow (OF) was applied continuously for a month, with an average flow of 3 L/s. Information about nutrient concentrations and properties of the effluent are shown in Table 1.

Five soil sampling plots were set up randomly in each study zone at ~1.5 m from the stream border, and measures were taken always at the same plots. Soil cores were extracted at each plot and samples were separated into 0–5 cm and 5–20 cm, because these depths corresponded to the most organic horizons and they potentially have the highest biological activity. One campaign was performed in the beginning of April 2019, before the wastewater effluent OF application, and another campaign was performed in May 2019, right after one month of the OF continuous application. In September 2020, before the beginning of a new Mediterranean hydrologic year, another campaign was performed to assess the recovery of soil properties and nutrient concentrations. The accumulated precipitation after the OF application and before the recovery sampling was 1095,3 mm (Catalan Meteorological Service).

2.3. Soil sampling and processing

To determine the soil properties and classification, 9 pits were dug in the study zones. Samples of every observable horizon were taken. For each horizon, samples were analyzed for granulometry, pH, electrical conductivity (EC), effective cations exchange capacity (ECEC), exchangeable sodium percentage (ESP), and organic carbon percentage (OC). Sieves were used to separate coarse fragments (>2 mm). An organic matter digestion was applied to the fraction with a diameter smaller than 2 mm. After the digestion, the samples were dispersed in an aqueous suspension and then, the pipette method (Robinson, 1922) was used to get samples of the different size particles: coarse sand (0.2–2 mm), fine sand (0.05–0.2 mm), coarse silt (0.02–0.05 mm), fine silt (0.002–0.02 mm) and clay (<0.002 mm) (Burt, 2004). To determine pH a pH-meter (Crison microPH 2002) was used in a suspension of 10 g of soil sample in 25 mL of distilled water (van Reeuwijk, 2002). Electrical conductivity (EC) was obtained from an aqueous suspension of 10 g of sample in 50 mL of distilled water (shaken and filtered with Whatman #42) using an EC meter (Crison microcm 2200) (Burt, 2004). To calculate

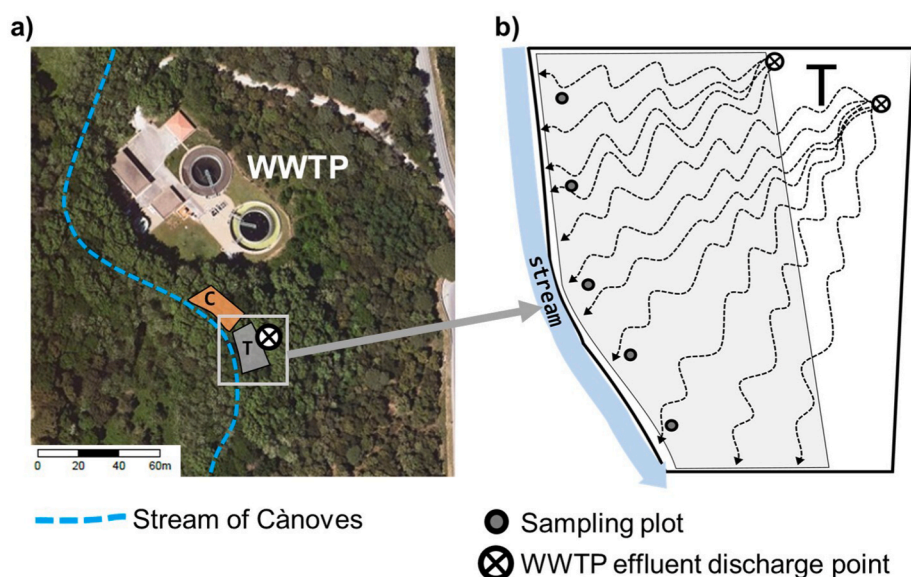


Fig. 1. a) Orthophoto (scale 1:5000) of the area of the WWTP of Cànoves i Samalús (Institut Cartogràfic i Geològic de Catalunya), modified to include the effluent discharge points and the studied zones control (C, in orange) and treated (T, in grey). b) Design of the treated zone sampling plots.

Table 1

Mean values \pm standard deviation of dissolved organic carbon (DOC), ammonium-N ($\text{NH}_4\text{-N}$), nitrate-N ($\text{NO}_3\text{-N}$), soluble reactive phosphorus ($\text{PO}_4\text{-P}$), electrical conductivity (EC), sodium (Na), pH and temperature (T) of the effluent of the WWTP.

Parameter	Mean \pm SD
DOC (mg/L)	6.74 ± 2.43
$\text{NH}_4\text{-N}$ (mg/L)	2.97 ± 0.32
$\text{NO}_3\text{-N}$ (mg/L)	0.23 ± 0.17
$\text{PO}_4\text{-P}$ (mg/L)	10.42 ± 3.59
EC ($\mu\text{S}/\text{cm}$)	983.28 ± 63.86
Na (mg/L)	130.48 ± 37.77
pH	7.22 ± 0.28
T ($^{\circ}\text{C}$)	24.75 ± 1.45

the effective exchange cation capacity (ECEC) and the exchangeable sodium percentage (ESP) a cation (Ca^{2+} , Mg^{2+} , K^{+} , Na^{+} and Al^{3+}) extraction with ammonium chloride 1M was done (Sumner and Miller, 2018). The extractions were analyzed in the Scientific and Technologic Services of Universitat de Barcelona using the ICP-MS technique. The OC content was determined by the Walkley and Black titration method (Mebius, 1960).

Soil samples were collected during the experiment at the sampling plots with a probe of 25 cm depth and separated into two depths (0–5 cm and 5–20 cm). At the laboratory, pH, EC and ESP were analyzed as described above. The soil organic matter percentage (SOM) was obtained by ignition of dry samples for 4 h at 450°C (Rowell, 1994). The content of ammonium (N-NH_4^{+}), nitrate (N-NO_3^{-}) and dissolved organic carbon (DOC) were also measured. These parameters were measured in an extract of 5 g of soil with 50 ml of 2.0 M potassium chloride (Keeney and Nelson, 1982). The salicylate-nitroprusside method (Baethgen and Alley, 1989) was used to determine ammonium content with a spectrophotometer (PharmaSpec U-1700 SHIMADZU). Nitrate was measured using a Technicon Autoanalyzer (Technicon, 1977) by the cadmium reduction method followed by a color development with sulfanilamide and naphthylethylenediamine (Keeney and Nelson, 1982). Dissolved organic carbon was analyzed with a Shimadzu TOC-V analyzer (SHIMADZU CSH)(Jones and Willett, 2006). Total phosphorus (TP) was measured in a soil extract of 0.5 g of soil in 20 mL of sulfuric acid by the colorimetric method of ammonium vanadomolybdate (Kuo, 1996) with

a spectrophotometer (PharmaSpec U-1700 SHIMADZU). Available phosphorus (P) was measured by the Olsen and Sommers (1982) in a soil extract 1:20 of NaHCO_3 0.5 M, using a spectrophotometer (PharmaSpec U-1700 SHIMADZU).

2.4. Data analysis

To determine the impact of the overland flow on soil properties and nutrients we used a BACI design (Green, 1979). According to this design, a significant interaction of the factors Period \times Zone would indicate a significant impact attributable to the treatment we are applying. Data were fitted to a linear mixed effects model using the restricted maximum likelihood method, where Period and Zone were fixed factors and Plot was a random factor nested within Zone to account for repeated measures. This model was fitted separately for each depth (0–5 and 5–20 cm). To evaluate the significance of the interaction of Period \times Zone, the degrees of freedom of the test were corrected by the Satterthwaite's method using the lmerTest R package (v3.1.3)(Kuznetsova et al., 2017). The ammonium concentration in 5–20 cm depth was log-transformed to satisfy the normality assumption. Finally, to determine if there were differences between zones after 17 months of the experiment, a one-way ANOVA was performed with the data of the last campaign. All statistical analyses were conducted in R version 4.1.0 (R Core Team, 2022).

3. Results

3.1. Soil characterization and classification

The physicochemical properties of the soil are presented in Table 2. The soils

had a neutral pH (mean \pm SD: 7.14 ± 0.44). The electrical conductivity values were low, comprehending a rank between 0.05 and 0.42 dS/cm with the highest values in the upper horizons of the soil profiles. Sandy loam and loamy sands texture classes were observed in the topsoil and subsoil, respectively, with an average of $72.85 \pm 14.19\%$ sand content and a clay content of $12.30 \pm 5.60\%$ (Fig. 2). The high standard deviation of the averages showed great variability in the content that varied substantially with depth. Generally, the soils contained a high proportion of gravels, which increased in depth, due to the fluvial origin of the C horizons. All the soils have more than 1% of soil organic carbon in the fine earth fraction to a depth of 50 cm from the mineral soil

Table 2

The horizons sequence, the organic carbon percentage as a weighted average to a depth of 50 cm (OC₅₀%) and the soil classification of each of the nine profiles (P).

P	Horizons sequence	OC ₅₀ %	Soil Classification
P1	A, AB, 2C	1.65	Eutric Skeletic Fluvisol (Arenic, Humic)
P2	A, B, 2C, 3C		Eutric Fluvisol (Arenic, Humic)
P3	A, B, 2B, 3C, 4R, 5R		Eutric Cambisol (Loamic, Humic, Raptic)
P4	A, B, 2BCE, 2C	1.27	Fluvic Skeletic Eutric Cambisol (Loamic, Humic)
P5	A, AB, 2C, 3C	1.09	Skeletic Eutric Fluvisol (Arenic, Humic)
P6	A, B, 2C, 3C, 4C	1.30	Eutric Fluvisol (Arenic, Humic)
P7	A, 2AC, 3AC, 4C, W	1.69	Skeletic Eutric Fluvisol (Arenic, Humic)
P8	A, 2B, 3C, 4C, W	0.48	Fluvic Skeletic Eutric Cambisol (Arenic, Humic)
P9	A, AB, C, 2B, 3B, 4C	1.41	Eutric Fluvisol (Loamic, Humic)

surface and evident stratification.

According to the World Reference Base (IUSS Working Group WRB, 2022), the studied soils were classified into two Reference Soil Groups: Fluvisols and Cambisols. Fluvisols are characterized by little differentiation of the profile and stratified fluvial material that reflects their depositional history. Cambisols have also been characterized by having little differentiation of horizons, but with the presence of a cambic horizon that begins in the first 50 cm of the soil, a lithic discontinuity, and more than 1% of soil organic carbon in the fine earth fraction to a depth of 50 cm from the mineral soil surface. Fluvisols in our samples were classified as Eutric Skeletic Fluvisol (Arenic, Humic) and Eutric Fluvisol (Arenic, Humic). Cambisols were classified as Eutric, Skeletic, Fluvic Cambisol (Loamic, Humic) and Eutric Cambisol (Loamic, Humic).

3.2. Impact of the WWTP effluent overland flow on soil properties

The effluent overland flow had a statistically significant impact on some physical and chemical soil properties, with generally, but not always, similar impacts in the studied depths (Fig. 3). Additionally, there was not a significant effect on EC in groundwater (Appendix A). Regarding soil environmental conditions (Table 3), there was not a statistically significant effluent effect on the topsoil moisture percentage, although it was higher in the treated zone. The topsoil temperature on the treated zone increased $\sim 3^\circ\text{C}$ after the effluent application, while it only increased $\sim 1^\circ\text{C}$ in the control zone. There was a statistically significant impact on topsoil temperature ($p < 0.0000$). The organic matter content (SOM) changed in both zones similarly, i.e. with no statistically significant interaction, decreasing in both depths (Fig. 3a). The electrical conductivity (EC) increased during the study period in the control zone, particularly in 5–20 cm depth. The treated zone had a slight increase in EC in 5–20 cm depth but there was not a statistically significant effluent effect on EC at any depth (Fig. 3b). The exchangeable sodium percentage (ESP) in the control zone had very similar values in both periods and depths. While in the treated zone the ESP before the effluent application was $1.49 \pm 0.29\%$ (mean \pm SE) in 0–5 cm and $1.30 \pm 0.14\%$ in 5–20 cm, and after it, the values became $3.77 \pm 0.54\%$ and $3.06 \pm 0.74\%$, respectively (Fig. 3c). There was an increase of about 200% in the treated zone, that resulted in a statistically significant impact of the effluent in ESP in both depths ($p < 0.0000$; $p = 0.0002$). The pH values were similar in all zones and periods, with mean values of 7.47 ± 0.03 and 7.66 ± 0.02 in the control and the treated zone, respectively (Fig. 3d), with no statistically significant impact in any of the depths.

Seventeen months after the end of effluent OF, we only found one statistically significant difference out of 8 tests (four variables, two depths). The SOM content had a similar depth pattern in both zones, with higher content in 0–5 cm than in 5–20 cm. It was higher in the

treated zone but there was not a significant difference between zones at any depth (Fig. 3a). The EC did not show a statistically significant difference between zones in the 0–5 cm depth in the recovery period. In 5–20 cm, however, the treated zone had higher values than the control zone ($p=0.0411$) (Fig. 3b). The ESP presented higher values in the 0–5 cm depth than in 5–20 cm depth in both zones. The values were slightly higher in the treated zone, but there was not a significant difference between zones at any depth (Fig. 3c). The pH values were very similar in both zones, and we found no significant differences between them at any depth (Fig. 3d).

3.3. Impact of the WWTP effluent overland flow on soil biogeochemistry

The WWTP effluent OF had an effect on the nitrate and ammonium content of the soil, with differences in relation to soil depth. The DOC concentrations fairly increased in the 0–5 cm depth of the treated zone although there was not a statistically significant effect in any of the depths (Fig. 4a). The TP concentration was quite similar among zones. There was not a statistically significant effect of the treatment at any depth (Fig. 4b). The available P was higher in the treated zone than in the control both periods before and after, and for the two depths. There was not a statistically significant effect of the treatment at any depth (Fig. 4c). The concentration of nitrate-N in soil increased in the control zone and it strongly decreased in the treated zone. There was a statistically significant effluent effect in both depths ($p < 0.0000$; $p=0.0008$) (Fig. 4d). The concentration of ammonium-N in 0–5 cm depth of the treated zone before the effluent application was $23.18 \pm 4.54 \text{ mgNH}_4\text{-N/kg}$ soil and after it, the mean value was $39.56 \pm 8.46 \text{ mgNH}_4\text{-N/kg}$ soil. Although the values had a high variance, there was a statistically significant effluent effect in 0–5 cm depth but not in 5–20 cm ($p = 0.0291$; $p=0.9460$) (Fig. 4d).

After 17 months of the effluent OF application, only the ammonium concentration differed between zones, and only in the 5–20 cm depth. Regarding DOC content, there were not significant differences between zones at any depth (Fig. 4a). The TP concentration was quite similar among zones and depths, and the highest values were found in 5–20 cm depth, with $244.43 \pm 20.76 \text{ mg P/kg}$ soil in the control and $278.91 \pm 18.46 \text{ mg P/kg}$ soil in the treated zone. There was not a statistically significant difference between zones at any depth (Fig. 4b). The available P concentration was higher in the treated zone than in the control and there were statistically differences between plots in both depths ($p = 0.0217$; $p = 0.0013$) (Fig. 4c). In the treated zone, in 0–5 cm depth, the mean value was 26.18 ± 5.89 and in 5–20 cm depth, it was 22.42 ± 3.66 . However the values of the treated zone during the recovery period were lower than the values before the experiment (36.19 ± 5.37 and 25.54 ± 2.91 , respectively for each depth). The concentration of nitrate-N in soil was quite similar in both zones and there was not a significant difference at any depth (Fig. 4d). The concentration of ammonium-N did not present a significant difference between zones in 0–5 cm depth. In 5–20 cm depth, there was a significant difference in ammonium-N content between zones; it was higher in the treated zone ($25.71 \pm 3.30 \text{ mg NH}_4\text{-N/kg}$ soil) than in the control ($14.59 \pm 2.11 \text{ mg NH}_4\text{-N/kg}$ soil) ($p=0.0217$) (Fig. 4e).

4. Discussion

4.1. Soil properties, profile characteristics and soil formation related to WWTP effluent overland flow

The two Reference Soil Groups found in the study site were Fluvisols and Cambisols. On one hand, Fluvisols are frequently found on past and present riverbeds in the Mediterranean region, including the east of the Iberian Peninsula, where our study site is located. Fluvisols were characterized by having little differentiation of the profile and a fluvial stratification manifested by the presence of fluvial material. One specific ecosystem service of Fluvisols is the ability to retain water but also trace

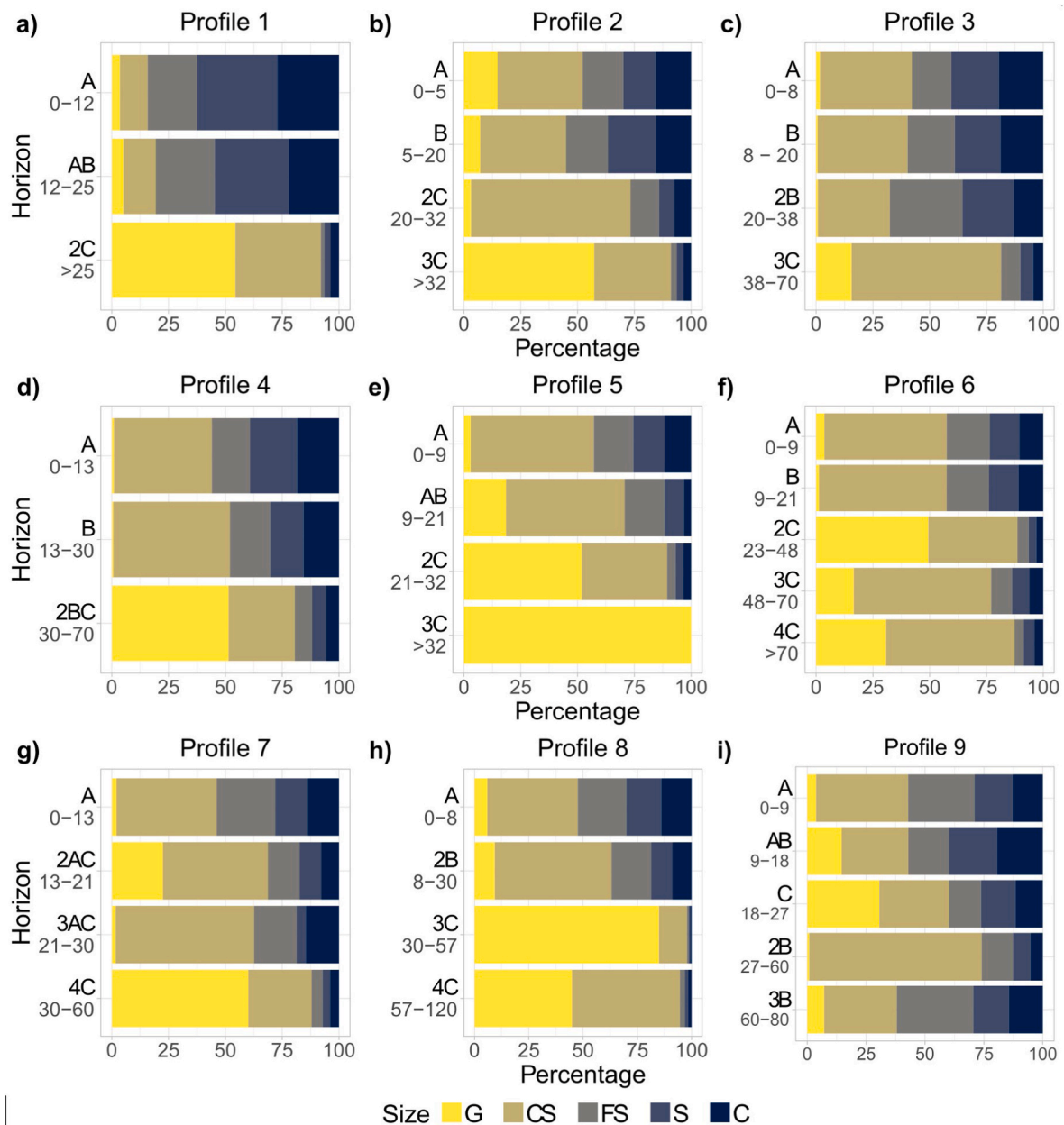


Fig. 2. Bar plot representation of the different particle size classes percentages (G: gravel, CS: coarse sand, FS: fine sand, S: silt, C: clay) for every horizon of the nine profiles (a-i), with the depth in cm.

or deleterious elements because of high content in soil organic carbon (humic qualifier). However, the coarse texture characteristics in one or more layers render them a high permeability but a low storage capacity for water and nutrients (arenic qualifier). On the other hand, Cambisols have been reported to be the most widely distributed soils of the Mediterranean region in different climatic areas. Furthermore, the Eutric Cambisol is one of the major units, which also is found in our study site region (Zdruli et al., 2010). All cambisol qualifiers show a great influence of the characteristics of fluvial deposits with evident stratification (Fluvic and Humic qualifiers) with a little soil formation after deposition and indicating an irregular decrease in soil organic carbon content with depth. These characteristics determine the dynamics of water and nutrients provided by effluents along the soil profile.

Therefore, the type of soil found in the riparian forest of Cànoves i Samalús is typical of this kind of ecosystem in the Mediterranean region. Soils developed within alluvial environments are highly heterogeneous, both in temporal and spatial scales, due to frequent redistribution of

sediments, organic matter and other materials (Naiman et al., 2005). This heterogeneity can be found in the soil profiles described in the study zone. It is especially remarkable the particle-size diversity, which favors the variability of soil moisture conditions, redox potentials, vegetation and temperatures, which in turn favors the occurrence of different biogeochemical hot spots (Vidon et al., 2010). Consequently, the soil of the present study has a great potential to receive the discharge of the WWTP effluent overland flow and retain and/or transform nutrients.

4.2. Impact of the wastewater effluent overland flow on soil properties

In general, the effluent OF did not have an important impact on soil properties except for exchangeable sodium percentage, which increased in the treated zone. Seventeen months after the OF application, there were not many significant differences between the control and treated zones.

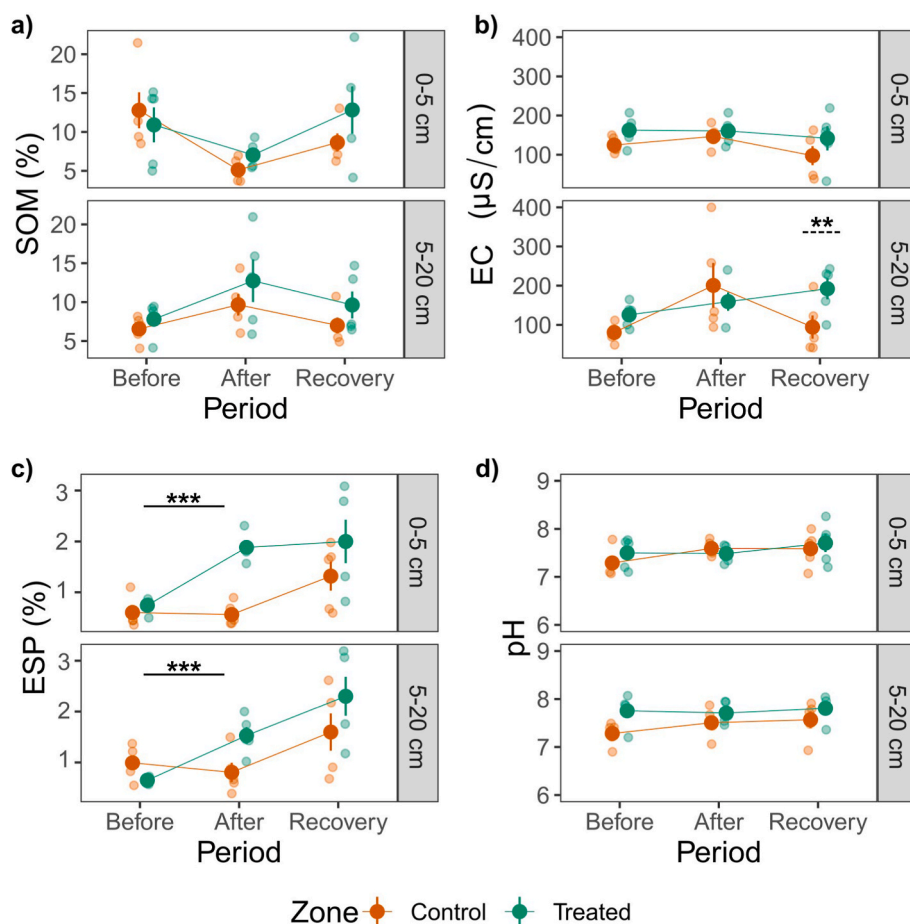


Fig. 3. Representation of the soil organic matter percentage (a), electrical conductivity (b), pH (c) and the exchangeable sodium percentage (d) in soil for the different sampling periods (before, after and recovery) and zones (control and treatment) for each depth (0–5 cm and 5–20 cm). The bold points represent the mean value and the bars represent the standard error. Significant interactions between factors period and zone of the BACI comparison are represented with ‘*’ above a straight line. Significant differences between zones in the recovery period are represented with ‘**’ above a discontinuous line. * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$.

Table 3

Mean value \pm standard error of temperature ($^{\circ}\text{C}$) and soil moisture percentage for topsoil in each period and zone.

Period	Plot	Temperature	Moisture
Before	Control	11.03 ± 0.15	32.54 ± 3.15
	Treated	11.50 ± 0.15	41.32 ± 3.46
After	Control	12.06 ± 0.22	34.18 ± 3.63
	Treated	14.90 ± 0.17	48.76 ± 4.92

The temperature increased in the treated zone because of the high temperature of the effluent ($\sim 25^{\circ}\text{C}$). The organic matter content was not affected by the effluent overland flow as expected, since the application period was just one month. A similar experiment in 2007 (Wen et al., 2007) also did not observe a change in SOM one month after an overland flow application, but they did observe an increase after one year. The presence of wastewater limits the availability of oxygen, thus aerobic decomposition may be restricted and in a longer-term application, we could expect an increase in SOM as many authors have reported (Walker and Lin, 2008; Tzanakakis et al., 2011; Jueschke et al., 2008).

The effluent OF did not have a significant impact in EC at any depth possibly because of the short-term nature of the application and the sandy textures of the soil, which facilitate water circulation and promotes salt leaching (Weil and Brady, 2017). Other studies did not report a significant difference in EC at the beginning of a wastewater irrigation period on agricultural soil (Morugán-Coronado et al., 2011). Significant increases in soil EC have been reported in numerous studies of long-term

wastewater irrigation systems and attributed to the high salt content of wastewaters (Singh, 2021). In relation to the impact in depth, there was a small increase of EC in 5–20 cm, which suggests that salts could accumulate in subsoil because of vertical transport. Duan et al. (2010) reported a significant increase in subsoil but not in topsoil, and Klay et al. (2010) reported a higher increase in EC in relation to soil depth. Regarding the ESP, there was a significant increase in both depths caused by the adsorption of the sodium present in the effluent. This impact was unexpected as other authors reported no increases in sodium in short-term wastewater applications in soils (Duan et al., 2010; Klay et al., 2010; Hussain et al., 2019). Considering the high load of effluent that was applied in this study (3 L/s in a $\sim 150\text{ m}^2$ surface), our results seemed to be more comparable to those of longer wastewater application in soils, in which significant increases in sodium soil content have been reported (Tzanakakis et al., 2011; Morugán-Coronado et al., 2011; Hussain et al., 2019; Sparling et al., 2006). However, the values of ESP resulting from the experiment are far from a sodic threshold (Weil and Brady, 2017). Despite the increase in ESP, the pH did not change in the treated zone at any depth, as expected, due to the high intrinsic soil pH buffer capacity and the small concentration of sodium on the cation exchange complex (Weil and Brady, 2017). These results agree with those of Duan et al. (2010), who did not find any significant change in soil pH at any depth in a short-term study of wastewater land application in a grass field. Nevertheless, other authors found a significant increase in soil pH in one- and two-year wastewater irrigation experiments (Sparling et al., 2006; Singh et al., 2012), also in long-term irrigations, which suggested that the soil might have adsorbed cations such as Na, K,

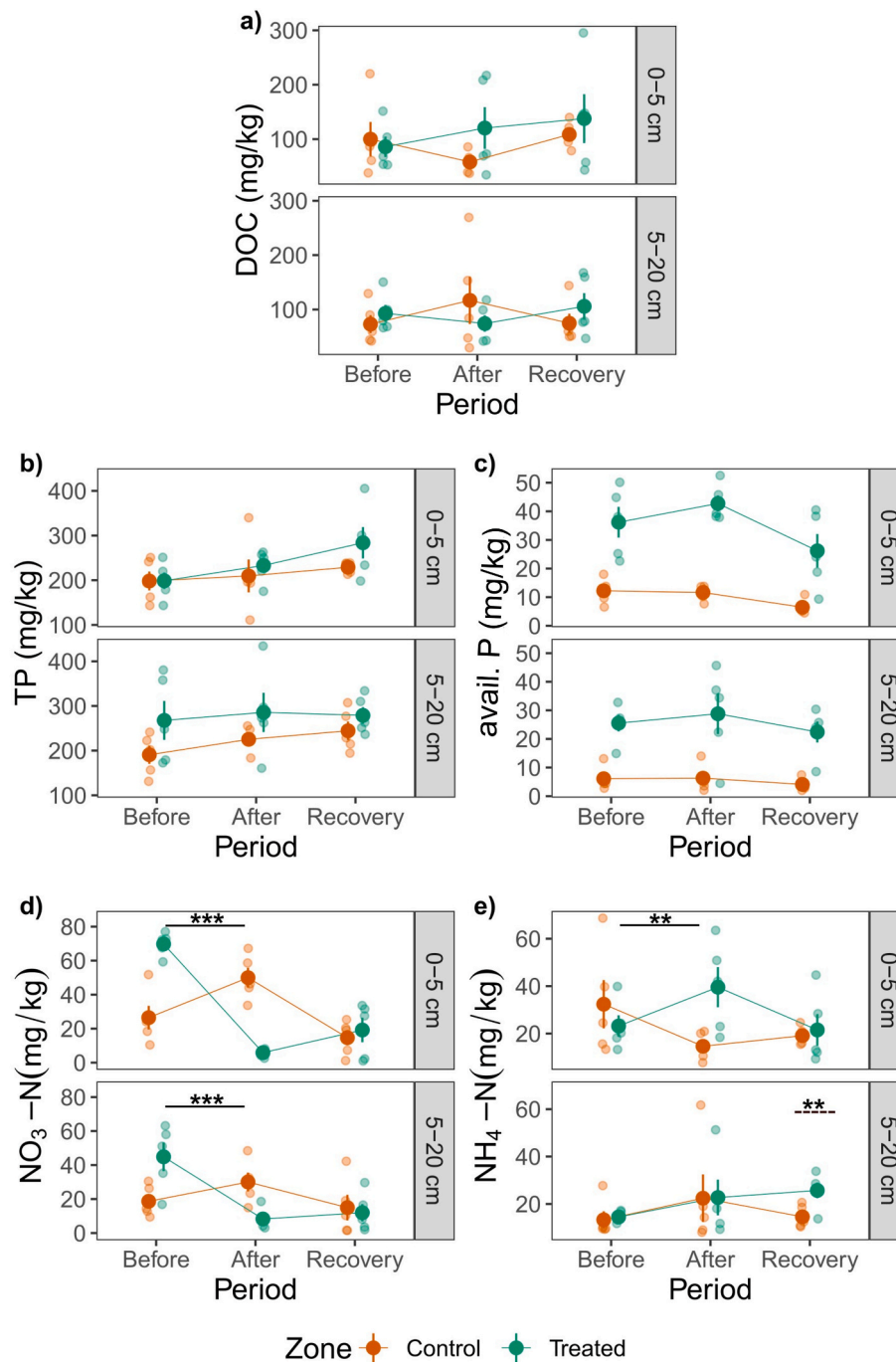


Fig. 4. Representation of dissolved organic carbon (a), total phosphorus (b), available phosphorus (c), nitrate-N (d) and ammonium-N (e) in soil for the different sampling periods (before, after and recovery) and zones (control and treated) for each depth (0–5 cm and 5–25 cm). The bold points represent the mean value and the bars represent the standard error. Significant interactions between factors period and zone of the BACI comparison are represented with ‘*’ above a straight line. Significant differences between zones in the recovery period are represented with ‘*’ above a discontinuous line. * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$.

Ca and Mg present in the wastewater (Hu et al., 2005).

After 17 months of the OF application, the EC in the 5–20 cm depth of the treated zone had significantly higher values than the control zone, unlike right after the experiment, when a small increase was appreciable, but not significant. This result indicates that there was an accumulation of salts in 5–20 cm depth, in agreement with what other authors have found (Duan et al., 2010; Klay et al., 2010). It is not clear that this increase is due to the effluent application because the values of EC were already slightly higher in the treated zone than in the control zone. Moreover, the recovery campaign was carried out after summer, with very low rainfall over the previous three months, and salts usually

get naturally concentrated as sodium did in both zones.

4.3. Impact of the wastewater effluent overland flow in soil biogeochemistry

Regarding nutrient concentrations, the OF had an effect on nitrogen compounds, especially in 0–5 cm depth. In general, the results that we found agreed with those found by Kumwimba et al. (2021), who demonstrate the effectiveness of a surface flow constructed wetland in decreasing nutrients loads from wastewater.

The effluent OF did not affect DOC, TP nor available P. Dissolved

organic carbon did not have a significant effect despite the DOC supply of the effluent because the application period was short. This agrees with Kayikcioglu (2012), who also did not report significant differences in DOC in a short-term study. Although not statistically significant, DOC showed a substantial increase in the 0–5 cm depth of the treated zone, which suggests that DOC could accumulate in a long-term application (Jueschke et al., 2008; Singh et al., 2012) and that this increase could be especially important in the topsoil (Klay et al., 2010). Although sedimentation and sorption processes in riparian buffers mainly control retention of TP (Hoffmann et al., 2009), we found no increase in TP content, as expected because of the short-term nature of the experiment. Besides, Baldovi et al. (2021) reported macrophytes assimilation as the major process in reducing the TP in vegetated surface flow constructed wetlands, dismissing other processes such as soil sorption. There was not an impact of the effluent on soil available P neither in the treated zone, since it was developed during the spring, when vegetation uptakes P for the active growth (Wen et al., 2007). Furthermore, Barton et al. (2005) found that effluent application to different soil types increased plant growth in comparison with unirrigated soils, and also reported an increase of N and P plant uptake.

Nevertheless, although both study zones are adjacent, available P in the treated zone was already higher than in the control zone before the experiment. This could be attributed to a different proportion of woody/herbaceous vegetation, which in turn can significantly affect the P uptake and should be taken into consideration for future studies (Hoffmann et al., 2009).

As expected, there was a significant decrease in nitrate-N in the treated zone in both depths. The main processes of nitrate-N transformation or removal in constructed wetlands are denitrification and plant and microbial uptake (Vymazal, 2007), and the conditions created in this study may have favored both processes, since soil moisture and temperature increased. The fact that denitrification could have occurred in the present study is supported by the results of Barbagli et al. (2019), who reported a decrease in nitrate concentration of up to 90% in a treated wastewater flow passing through a sandy loam soil in an experiment of soil columns, which was attributed to heterotrophic denitrification.

The concentration of ammonium-N increased in the 0–5 cm depth of the treated zone because the effluent had a very high ammonium concentration. Tzanakakis et al. (2011) reported higher N concentrations in topsoil after a wastewater effluent land application. Ammonium is positively charged and can readily adsorb to negatively charged clay particles and organic matter present in soil surface. However, the increase was slight, and it did not occur in 5–20 cm depth, suggesting that part of it might have been removed. Ammonium uptake by plants could have played an important role in this removal since it is preferred over nitrate as a source of nitrogen for assimilation (Kadlec and Wallace, 2009). Another mechanism that could have been at play was nitrification coupled with denitrification, which has been reported to be the major removal process in many treatment wetlands (Vymazal, 2007). In a vegetated buffer zone (Koenig and Trémolières, 2018), a removal of 69% of ammonium by partial nitrification and denitrification was estimated. In our experiment, this path could have been enhanced by the heterogeneous soil texture and microtopography, both of which facilitate a diversity of redox conditions. In the recovery period, it is remarkable that the concentration of nitrate increased in the 0–5 cm depth of the treated zone and had values like those found in the control zone, so the effluent just caused reduced oxygen concentrations that enhance denitrification while it was applied, as it was observed by Li et al. (2014). While after the experiment ammonium increased in the 0–5 cm depth of the treated zone, after 17 months of the experiment, it was higher in the 5–20 cm depth instead. This result could be related to the higher SOM in the 5–20 cm depth of the treated zone, which could support higher ammonification rates. Riparian soils are very heterogeneous in biogeochemical characteristics so these differences can be explained by the presence of microzones with more accumulation of

SOM or with more limiting ammonium transformation potential (Naiman and Décamps, 1997; Naiman et al., 2005). In the recovery period, the available P was higher in the treated zone than in the control zone, but it was also higher before the experiment. Furthermore, the values in both zones were lower than before the WWTP effluent overland flow, thus we can not conclude that it was affected by the effluent.

Although our study was mainly focused on the immediate impacts on soil properties and biogeochemical compounds, further research should address some environmental impacts. In this sense, other studies regarding WWTP effluents application on soils, found that the high salinity of the effluents decreased the basal soil respiration (Morugán-Coronado et al., 2011). As well as other studies found negative effects when sodium, salts, and nitrates reached groundwater (Duan and Fedler, 2007; Jalali et al., 2008). Moreover, other important parameters regarding microbiological contaminants should be monitored before a long-term application (Schierano et al., 2020). However, other works showed the potential positive effect on macrophyte development (Tzanakakis et al., 2009; Lavrnić et al., 2020).

4.4. Conclusions

This study is a first approximation to evaluate the potential of riparian soils to reduce the nutrients from WWTP effluents. Although significant impacts have occurred, none of them constituted a damage to the environment. Moreover, the results provide useful information about the nutrient content, showing that riparian soils have a great potential to decrease nitrate from WWTP effluents. Further research is needed to assess more deeply this potential as well as the impacts in a mid- and long-term applications. Future research could include the study of effluent subsurface application to increase the residence time, and the operation with intermittency, so aerobic and anaerobic conditions could be alternated to enhance ammonium removal through nitrification-denitrification pathways.

CRediT authorship contribution statement

Laura Escarmena: Writing – original draft, Visualization, Supervision, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Núria Roca:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Joan L. Riera:** Writing – review & editing, Supervision, Methodology, Formal analysis, Data curation. **Teresa Sauras-Yera:** Supervision, Methodology, Investigation, Conceptualization. **Santi Sabaté:** Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization. **Francesc Sabater:** Writing – review & editing, Writing – original draft, Supervision, Resources, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix B. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jenvman.2024.121778>.

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