



# Vegetation Filters for brewery wastewater treatment: Results of a 'lab-to-field' procedure under Mediterranean conditions compared to a conventional WWTP

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## ABSTRACT

Water is a vital element in the brewing industry, being present in all facets of its operation. This industry generates substantial wastewater quantities, requiring intensive and expensive treatment in Wastewater Treatment Plants (WWTP). Vegetation Filters (VF) are a type of Nature-Based Solution (NBS) for wastewater treatment that offers a sustainable alternative to these WWTP. In this work, an innovative 'Lab-to-field' procedure including several trials at different scales was conducted to assess the applicability of VFs to the brewing industry, starting from trials under greenhouse conditions to a real scale pilot plantation under Mediterranean conditions (Spain). This represents the first attempt to assess the use of VF in the brewing industry. The occurring processes during the trials under controlled conditions match those observed on the field, thus validating the experimental procedure as a quick and simple tool for new scenarios. While total phosphorous (TP), chemical oxygen demand (COD) or total suspended solids (TSS), among other parameters, were easily removed from the wastewater, nitrogen (N) leaching towards the aquifer was observed, especially during the second period of the VF operation. This was caused by the detrimental effects of the high salinity (especially sodicity) on soil and plant health, highlighting the need for rigorous control and maintenance. Minimizing the sodium ( $\text{Na}^+$ ) amounts in influent wastewater and the addition of soil amendments and proper soil and cultural management practices would be most beneficial for the overall system performance. More research is needed addressing the effects of suggested limitations on  $\text{Na}^+$ -rich compounds during wastewater pretreatment

## 1. Introduction

Water is essential of the brewing industry, involved in nearly all its processes and operations. In modern breweries, water consumption typically ranges between 4 and 10 liters per liter of beer produced (Santonja et al., 2010). This variability hinges on factors like brewery scale and regional development, with figures below 4 L/L in historical companies within developed nations and

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surpassing 8 L/L in emerging regions (Heineken N.V., 2018; Zhao et al., 2018).

Beer production also generates significant amounts of wastewater, averaging between 3 and 6 L of wastewater for each liter of beer produced, roughly 70 % of total water usage (Brewers Association, 2014; Santonja et al., 2010). Brewery wastewater typically presents very high organic loads (sugars, soluble starch, ethanol, volatile fatty acids, etc.), and varying pH values and ionic concentrations, depending on the amount of yeast present in the effluent and the type of chemicals used in sanitizing and cleaning processes (Simate et al., 2011). The treatment of such wastewater usually requires, at least, the use of primary and secondary treatments, which incurs high energy cost and the use of chemical additives within brewery wastewater treatment plants (WWTP) before discharge to sewage networks or natural watercourses (Santonja et al., 2010; Simate et al., 2011).

Nowadays, there's a strong push towards Nature-Based Solutions (NBS) in order to reduce waste generation and enhance environmental sustainability within a circular economy framework, especially concerning water (European Commission, 2022, 2018). The International Union for the Conservation of Nature (IUCN) defines NBS as "actions to protect, sustainably manage, and restore natural or modified ecosystems, which address societal challenges effectively and adaptively, simultaneously providing human well-being and biodiversity benefits" and "initiatives that have simultaneously helped protect, manage and restore the environment while delivering tangible and sustainable benefits for people" (IUCN, 2020). In this sense, the search for sustainable and environmentally friendly solutions for wastewater treatment with economical or ecological benefits represents a clear case of NBS implementation in the industry.

Land application of wastewater has a long history, dating back to ancient Greece and peaking during the 19th century (Crites et al., 2006; Metcalf and Eddy, 2003). However, in the 20th century, as population growth and industrialization led to more complex water treatment needs, these systems were largely replaced by conventional treatment plants, except in smaller towns or areas with space constraints (Jewell and Seabrook, 1979). Today, contemporary trends towards environmental sustainability are driving the resurgence of these systems.

Vegetation Filters (VF) represent a form of NBS where pre-treated or treated wastewater is used to irrigate planted soils, typically forestry plantations. Through the synergistic action of soil, plants, and microbiota, pollutants in water undergo attenuation via sorption, plant uptake, and biodegradation processes, resulting in purified water that can recharge the underlying aquifer (de Miguel et al., 2014; Pradana et al., 2021). Groundwater recharge can be of great interest, as it stands as a primary water resource worldwide, exhibiting a remarkable resilience to climate variability, and being vital in the current climate change scenario and as a supplement to surface water bodies (Dillon et al., 2020; Taylor et al., 2013; UNESCO, 2021).

*Salicaceae* species, such as poplars or willows, are the most frequently used in these systems (Guidi Nissim et al., 2021; Pradana et al., 2021), due to their high evapotranspiration, growth, and nutrient uptake rates and their bioaccumulation capacity – properties particularly interesting in a phytoremediation context (Dipesh et al., 2015; Zalesny et al., 2019). Willows are more frequently used in temperate and cold climates (Aronsson et al., 2014; Guidi Nissim et al., 2013), while poplars are preferred in mediterranean areas, such as Italy or Spain (de Miguel et al., 2014; Guidi et al., 2008; Tzanakakis et al., 2012).

This study seeks to evaluate: i) the outcomes of a series of tests under controlled conditions, testing different *Salicaceae* plant material, focusing on their capacity to attenuate contaminants present in brewery wastewater and the utility of this procedure to reflect the occurring processes in a real case scenario; ii) the results of a pilot VF installed in the field next to a local brewery under Mediterranean conditions; and iii) the characterization of this brewery's wastewater, along with the geological characterization of the factory surroundings, as well as the impacts of the VF operation on them after 2 years. This work represents, to our knowledge, the first example of the application of VF to the brewing industry, as these systems have been typically tested in urban wastewater treatment. Thus, the insights gained from this study could help clarify the possibilities and limitations of VF. Moreover, our lab-to-field procedure (see below) could serve as a guideline for testing VF in new scenarios

## 2. Materials and methods

In this work, we developed a 'Lab-to-field' approach, progressing through a sequence of steps that transitioned from controlled laboratory experiments to a real-world pilot VF, irrigated with wastewater from the brewery's secondary treatment process. Insights from the controlled tests informed the VF design in the field, and the procedural framework encompassed the following stages:

- i) Plant material selection trial I: pre-screening in hydroponic culture under greenhouse conditions.
- ii) Plant material selection trial II: screening in substrate (pots) under greenhouse conditions.
- iii) Field plantation.

In each stage, the tested genotypes were selected based on their survival and development rates, their biomass production and their treatment performances, but also attending to their commercial availability and climatic suitability.

For the geological cartography of the area, PNOA aerial images and Digital Terrain Models from the Spanish National Geographic institute were used as base materials. Geological map units and cross-sections were created using QGIS v.3.22.7 and Adobe Illustrator CS6.

### 2.1. Plant materials

Details concerning plant material selection trials and field planting procedures are elaborated in Pradana et al. (2023) and summarized herein, where we focus on the evaluation of water pollutant attenuation capacity.

### 2.1.1. Hydroponic trials

23 genotypes of *Salicaceae* were selected for this study. These genotypes, including both productive (*P. x canadensis* Mönch 'I-214', 'MC', '2000 Verde', 'AF34', 'AF2', 'Guardi', 'Triplo', 'Luisa Avanzo', 'I-454/40', and 'Branagesi'; *P. x generosa* Henry x *P. trichocarpa* Torr. & A. Gray 'AF8'; *P. deltoides* W. Bartram ex Marshall 'Viriato'; *P. x generosa* Henry x *P. nigra* L. 'Monviso'; and *Salix matsudana* Koidz. x *Salix* spp. 'Levante') and autochthonous hybrids (*P. alba* L. autochthonous Guadalquivir River basin 'PO-10-10-20', 'PO-9-16-25', and 'GU-1-21-29'; *P. alba* L. autochthonous Jalón River basin 'J-1-3-18'; *P. alba* L. autochthonous Almanzora River basin 'S-18-5-22'; *P. alba* L. '111PK'; *Salix atrocinerea* Brot. Autochthonous Ebro River basin; *Salix alba* L. autochthonous Ebro River basin; and *Salix eleagnus* autochthonous Ebro River basin), have been extensively tested in Mediterranean climates. The hydroponic trial conducted under greenhouse conditions utilized all 23 genotypes, with subsequent selection based on tolerance and productivity criteria for further steps (Pradana et al., 2023).

### 2.1.2. Planted pots trials

For the selection trial in substrate under greenhouse conditions, 7 genotypes were evaluated: 5 from the *Populus* genus ('I-214', 'MC', '2000 Verde', 'AF34', and 'PO-10-10-20') and 2 from the *Salix* genus ('Levante' and *S. atrocinerea*). These genotypes were selected based on survival rates, ecophysiological activity, and biomass production (especially root development) during the hydroponic trials.

### 2.1.3. Field plantation

In the field planting phase, due to the increased space availability, 9 genotypes could be selected for planting, based on their performance during the previous trials, concerning both growth and biomass production and wastewater treatment capacity. 8 genotypes from the *Populus* genus ('I-214', 'MC', '2000 Verde', 'AF34', 'AF-8', 'Triplo', 'PO-10-10-20', and 'GU-1-21-29') and 1 from the *Salix* genus ('Levante') were selected. (Pradana et al., 2023).

## 2.2. Experimental design

### 2.2.1. Greenhouse trials

Both hydroponic and pot trials were carried out under controlled greenhouse conditions (max T:  $25 \pm 3^\circ\text{C}$  and min T:  $10 \pm 3^\circ\text{C}$ , humidity 65 % and lighting  $1000 \mu\text{E m}^{-2} \text{s}^{-1}$ ). Unrooted cuttings, measuring 30 cm in length, were selected from lignified one-year-old stems.

In the hydroponic trial, ten replicates per genotype were randomly installed in 55 L containers, inserted in a rubber board above the water level to prevent contact with the container. A 5 W pump was installed in each container to avoid stagnation. Five containers were filled with the brewery wastewater, and the other five with a control solution (tap water with a commercial nutrient solution containing (w/v) 8.2 % of free amino acids, 16.4 % of total amino acids, 5.8 % of total nitrogen (TN), 5.8 % of  $\text{P}_2\text{O}_5$ , 5.8 % of  $\text{K}_2\text{O}$ , 0.4 % of B, 0.34 % of Cu, 3.37 % of Fe, 2.02 % of Mn, 0.13 % of Mo, 0.47 % of Zn and 5.33 % of MgO (Daymsa, 2003; Upendri and Karunarathna,

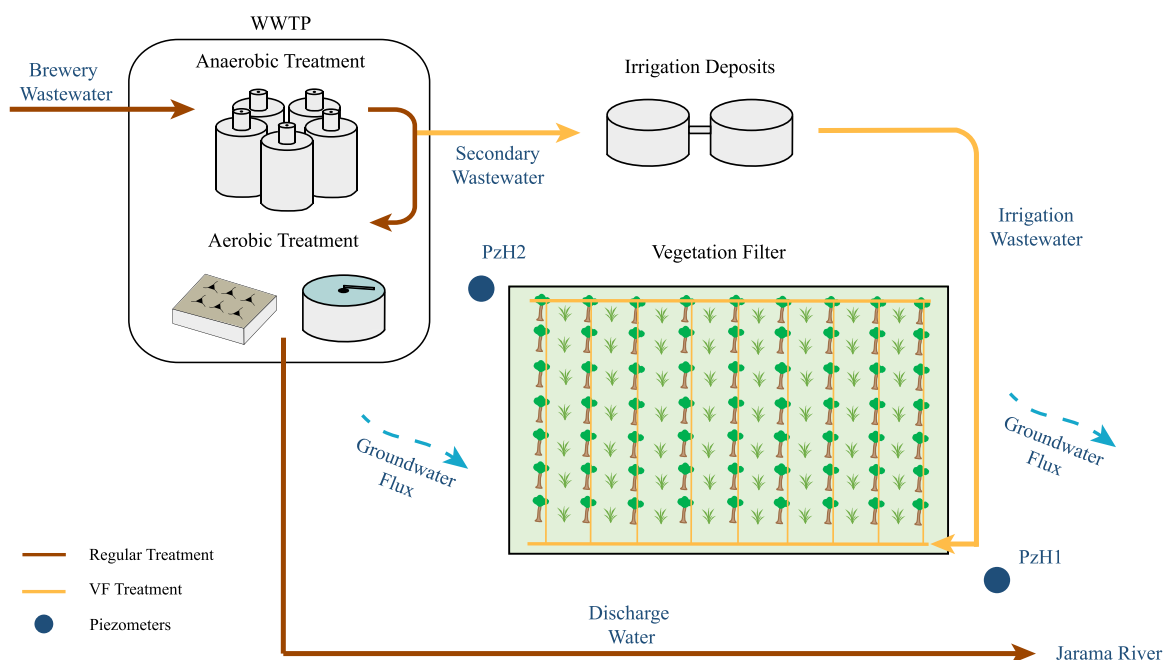


Fig. 1. Scheme of the Vegetation Filter on the WWTP surroundings.

2021) at a concentration of  $0.84 \text{ ml L}^{-1}$ . Both treatments were renewed weekly, and the trials were maintained for two months (64 days, March - April).

For the potted trial, ten replicates of each of the seven genotypes were individually planted in 15.5 L pots with a TKS-2 peat substrate and river sand mixed at a ratio of 3:1 and placed in a randomized layout. Five replicates of each genotype were manually irrigated with brewery wastewater, while the other five were equipped with an automatic irrigation system using tap water. The wastewater for irrigation was renewed and sampled weekly and the trial lasted for four months (March – June). Irrigation was performed manually and based on the water necessities measured using several humidity probes installed in different pots.

### 2.2.2. Field pilot plantation

In the field, a selection of the genotypes tested under controlled conditions was used to establish a  $1000 \text{ m}^2$  plantation in an industrial site adjacent to the Heineken beer factory in Madrid ( $40^{\circ}35'08.8''\text{N}$   $3^{\circ}34'18.8''\text{W}$ ) at a density of  $10,000 \text{ cuttings ha}^{-1}$  ( $2 \times 0.5 \text{ m}$ ). 60 % of the VF area was planted with these genotypes for further testing, while the remainder was planted using the poplar hybrid 'I-214', as it is a widely used genotype in the region and, particularly, in VFs treating urban wastewater.

Two piezometers were strategically installed within the vicinity of the VF to monitor aquifer dynamics and quality. One piezometer was installed upstream of the plantation to ensure its readings were not affected by the activity of the VF, while the second was installed downstream of the plantation.

A drip irrigation system was installed in the plantation using the secondary wastewater from the anaerobic reactor of the factory's WWTP (Fig. 1). Two tanks - a settling tank and a pumping tank - were installed, the latter equipped with an irrigation programmer, as well as a flow meter at the head of the plantation. The measured flow rate was  $200 \text{ L min}^{-1}$ , and irrigation timing was set according to the PET calculations for the area. Irrigation lines covered the whole plantation and were connected via two distribution lines, to achieve homogeneous irrigation.

Soil sampling was performed before plantation using a 16-point grid (10 m lengthwise, 5 m widthwise), with a composite sample created by mixing all 16 samples for characterization (Table 1). Additionally, 15 undisturbed soil samples were taken from 5 of the 16 points (3 cylinders per point) using a soil cylinder sampler (Eijelkamp equipments, the Netherlands). After two years of irrigation, the soil was resampled to evaluate potential impacts from the VF's operation. Given the soil's high variability, final samples were analyzed individually, both inside and outside the VF for comparison. Prior to the plantation, the area was tilled following the guidelines by

**Table 1**

Methodologies used in physicochemical characterization of the secondary wastewater, soil and climate characteristics of the field site. Presented values for climate and soil characteristics correspond to those analyzed prior to the VF installment and operation.

	Parameters		Methodology	Mean value and SD
Wastewater	pH		Electrometry UNE-EN ISO 10523:2008	
	EC	(mg/L)	Electrometry UNE-EN ISO 27888:1997	
	TN		Photometry UNE-EN ISO 11905-1:1998	
	TP		Photometry UNE-EN ISO 6878:2005	
	TOC		TOC analyzer	
	COD		Photometry UNE 77004:2002	
	TSS		Filtration UNE-EN 872:2006	
	$\text{NH}_4^+$		Ionic chromatography	
	$\text{Na}^+$			
	$\text{Cl}^-$			
	$\text{SO}_4^{2-}$			
	SAR		Calculated	
	MT	(°C)	Source: SIAR, Spanish Government	14.18
Climate	MMTW			33.42
	MMTC			-0.42
Soil	pH		UNE ISO 10390:2012	8.48
	EC ( $\mu\text{S/cm}$ )		UNE 77308:2001	172
	Clay	(%)	UNE 103102:1995	22.4
	Lime			31.5
	Sand			46.2
	Bulk density ( $\text{g/cm}^3$ )		Undisturbed core sampling	1.58
	Total N (mg/g)		Kjeldahl method	1.29
	Assimilable P (mg/g)		Spectrophotometry	64.8
	$\text{CaCO}_3$ (g/kg)		Bernard calcimeter	42.1
	$\text{Na}^+$	(mg/kg)	ICP-MS	93.8
	$\text{K}^+$			258
	$\text{Ca}^{2+}$			3665
	$\text{Mg}^{2+}$			539
	CEC (cmol/kg)			19.8
	Organic Matter (%)		LOI calcination	2.65

EC, Electric Conductivity; TN, Total Nitrogen; TP, Total Phosphorus; TOC, Total Organic Carbon; COD, Chemical Oxygen Demand; TSS, Total Suspended Solids; SAR, Sodium Adsorption Ratio

MT, annual mean temp.; MMTW, mean maxim temp. of warmest month; MMTC, mean min. temp. of coldest month; EC, Electric conductivity; TKN, total Kjeldahl nitrogen; CEC, Cation Exchange Capacity.

Sixto et al. (2010). During tilling, various solid wastes, including debris, glass, and diatomaceous earth from brewery filtration, were found, reflecting the industrial history of the site over decades and contributing to soil variability, particularly affecting hydraulic properties like permeability.

Climatic parameters were sourced from the Spanish Government's service SIAR (Agroclimatic Information for Irrigation Service). This service calculates the reference evapotranspiration (Eto) within an area using direct measurements of wind speed, air temperature, solar radiation, and humidity. Calculations are performed using a mixed formula based on the Penman-Monteith equation while integrating parameters and calculations of the Blaney-Criddle, Hargreaves and Radiation methods, following the FAO revision of crop water requirements methods (Allen and Pruitt, 1986; Smith et al., 1992).

### 2.3. Wastewater characterization

The secondary wastewater from the brewery was sampled weekly for its characterization, both during the trials under controlled conditions and operational phase of the VF, and following various methodologies based on ISO-UNE standards (Table 1). Results on wastewater characterization are later described in Section 3.2.

During the trials under controlled conditions, influent wastewater was sampled directly at the WWTP in the brewery, prior to its transport to the greenhouse in which the test was conducted. In the hydroponic trials, the outlet wastewater was collected from the containers after seven days in contact with the cuttings. During the planted pots trial, the effluent water after its infiltration through the substrate was sampled from each pot using a syringe, and a composed sample per genotype and treatment was prepared weekly for its analysis.

During the VF operation in the field trial, irrigation wastewater was weekly sampled from 5 m<sup>3</sup> the pumping tank at the start of the drip irrigation system, as well as groundwater from both piezometers.

In all cases, samples were collected in 1 L polyethylene bottles and transported to the lab for their analysis.

### 2.4. Data analysis

Data analysis and visualization were performed using the Statistical package Statgraphics 19X-64 and R software v.4.1.2. Sample projections onto the Piper diagram for water classification (Piper, 1944) and onto the USSL diagram for irrigation water adequacy (US Salinity Laboratory Staff et al., 1954) were performed using the R packages 'The soil texture Wizard' (Moeys, 2024) and 'Iwaqr' (Ali, 2024), respectively.

Percentages of removal efficiency for each parameter were calculated using the input and output values (influent and effluent water) using the Eq. (1), similarly to Worku et al. (2018). The selected parameters were the main macronutrients N, P, K, Ca, Mg, and sulfur, as well as other parameters related to the organic load (Chemical oxygen demand -COD-, Total organic carbon -TOC- and salinity (Electric conductivity -EC-, Na<sup>+</sup>, Cl<sup>-</sup>).

$$\text{Removal efficiency} = \frac{(C_{\text{inflow}} - C_{\text{outflow}})}{C_{\text{inflow}}} * 100 \quad (1)$$

When evaluating the VF performance in the field, ANOVA was used followed by Duncan's mean separation test to identify significant differences between influent wastewater for irrigation and groundwater collected upstream and downstream of the pilot plantation. This analysis was applied to each parameter separately, after confirming that normality was met, and distinguishing two periods: P1 (from VF installation to April 2022, including the first activity-inactivity cycle) and P2 (May 2022 - January 2023, covering the second activity-inactivity cycle). These statistical analyses were performed using the Statgraphics 19X-64 software.

A monthly water balance comparing the amount of water applied via irrigation or rain with the amount of water "lost" via the evapotranspiration of the plantation, using the climatological variables exposed above, and expressed in m<sup>3</sup> for each month.

## 3. Results and discussion

### 3.1. Geological setting and mapping

The study area is located near the north boundary of the Tajo Basin (Alcalá et al., 2004), which encompasses various continental Cenozoic detritic, carbonatic and evaporitic materials. Specifically, the brewery's WWTP is placed within the lowest and most recent level of fluvial terraces along the middle course of the Jarama river, after its progressive incision during the Quaternary, eroding the previous Neogene detritic materials in the surroundings (Supplementary Materials, SM, Fig-S1).

*In-situ* core logging during drilling the geological deposits in both piezometers to be intercalations of sands, silts, and clays, occasionally interlayered with cobble-rich levels. These lithologies are indicative of a dynamic fluvial deposition system characterized by fluctuating energy levels resulting in thin levels with limited lateral continuity and lateral facies changes. Both piezometers exhibit rapid recovery rates, with the water table swiftly stabilizing within a few minutes after pumping (SM, Fig-S2). This can be attributed to the site being in the discharge zone of the aquifer, being right next to the Jarama river and featuring a shallow water level.

In general, the geological setting seems to be quite appropriate for the implementation of a VF, as it is compound of detritic porous materials, with the absence of large impermeable layers and the existence of an accessible underlying aquifer.

### 3.2. Wastewater characterization and treatment

The chemical characterization of the brewery's secondary wastewater shows, overall, tolerable pH values for poplar irrigation. However, it exhibits high electric conductivity and sodium adsorption ratio (SAR) values, due to elevated  $\text{Na}^+$  and  $\text{Cl}^-$  concentrations. Indeed, this secondary wastewater can be classified as sodium bicarbonate type using Piper classification (Piper, 1944) and within the C4-S4 class, using the USSS classification, making it inappropriate for irrigation (US Salinity Laboratory Staff et al., 1954) (SM, Figs-S3-S4). Despite this and given the interest in testing the VF application to this kind of scenario, we decided to develop the trials with no additional pretreatment. Notable amounts of nitrogen (mostly in the form of  $\text{NH}_4^+$  and organic nitrogen) are also present, while total phosphorous (TP) values are within the typical range for urban wastewater, which, from our experience, should not present a significant issue as P is easily removed from wastewater when used for VF irrigation.  $\text{SO}_4^{2-}$  values are significantly lower than some natural waters, and far from being hazardous to the environment. We did not find any remarkable seasonal variability in wastewater composition, already pretreated and homogenized during the first stages of the WWTP.

Thus, the main concerns regarding wastewater composition are salinity and nitrogen concentration. The application of high amounts of these parameters can compromise the efficiency of the system, while also posing a risk to plant, environmental and human health. Leaching of nitrate towards groundwater can make it unfit for drinking purposes, and its presence in water bodies leads to eutrophication and anoxia, causing severe environmental damages (Bijay-Singh and Craswell, 2021). On the other hand, the toxicity effects of salt-rich waters ( $\text{Na}^+$  and  $\text{Cl}^-$ ) is also known, both for plants and humans (Deivayanai et al., 2024; He et al., 2023).

#### 3.2.1. Hydroponic trials

The attenuation percentages of contaminants for each analyzed parameter vary between trials, with generally higher levels observed in the 2nd trial and lower levels in the 1st trial (Fig. 2). However, consistent trends in attenuation rankings persist across trials. In both cases, organic matter (BOD, TOC, and COD) and total suspended solids (TSS) exhibit the highest attenuation percentages, followed by nutrients (TN and TP), and finally, by the remaining ions present in the wastewater.

Notably, oxidized ions such as  $\text{PO}_4^{3-}$  and  $\text{SO}_4^{2-}$  tend to display negative percentages (SM, TabS1), suggesting the presence of reduced species of these elements in the wastewater. Aeration of water likely enhanced the oxidation of these species, akin to conventional wastewater treatment plants (Drewnowski et al., 2019; Levy et al., 2011; Stare et al., 2007). This observation is supported by the higher (positive) attenuation percentages of  $\text{NH}_4^+$ ,  $\text{K}^+$ , and  $\text{Na}^+$  in the 2nd trial compared to the negative percentages observed in the 1st trial. We attribute these differences between trials to a minor change in the experimental setup. In the 1st trial, the foam containing the cuttings floated on the wastewater surface, while in the 2nd trial, it was positioned on two small wooden slats above the water surface. This change likely facilitated better aeration and oxygenation of the water, thereby improving the treatment performance for exchangeable cations and nitrogen (Aboutayeb et al., 2023; Wang et al., 2024).

Regarding nitrogen transformations, in the 1st trial, the predominant process appears to have been the mineralization of organic nitrogen into  $\text{NH}_4^+$ , whereas increased aeration in the 2nd trial facilitated more efficient performance of the anammox process by microbiota, leading to higher TN attenuation and the presence of minor but detectable amounts of  $\text{NO}_3^-$  in the wastewater via nitrification and denitrification processes.

Furthermore, the contaminant concentrations in the influent water exhibited a significant increase towards the latter stages of the 1st trial (SM, Figs-S5-S6). This alteration in the wastewater composition can be attributed to the irregular operations at the factory during the lockdown period imposed by the COVID-19 pandemic. Thus, the absence of this alteration during the 2nd trial makes it more reliable, as it reflects the influent wastewater characteristics more precisely.

In broad terms, the hydroponic trials demonstrated a reasonable attenuation capacity for certain analyzed parameters, particularly those associated with organic matter and suspended solids. Nevertheless, these attenuation percentages fell short of those attainable in a fully operational Vegetation Filter (Pradana et al., 2021). This variance in treatment efficacy can be ascribed to the absence of a soil

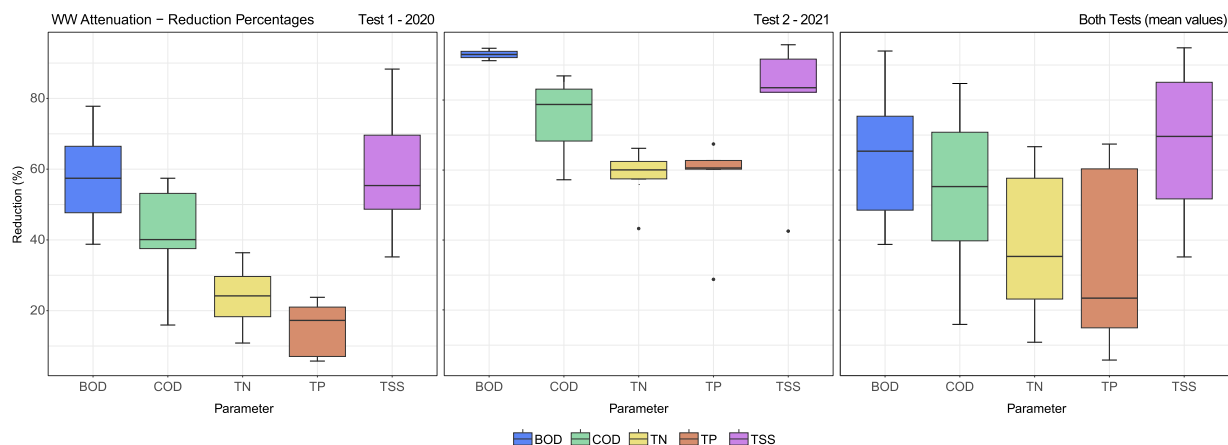


Fig. 2. Mean attenuation percentages for the main parameters during the hydroponic trials.



system, consequently lacking various attenuation mechanisms such as adsorption and cation exchange and limiting biodegradation processes. Thus, although the main objectives of these trials were to serve as a quick and easy method for testing a wide variety of genotypes and enhance selection criteria in further investigation (i.e. the planted pots trial and the field pilot plantation), it also provides quantitative evidence of the importance of a proper soil system presence in a VF in terms of water treatment capacity.

### 3.2.2. Planted pots trials

In the planted pots trial, wastewater treatment capacity was assessed for each of the genotypes (those that presented the most interesting behavior during the hydroponic tests) separately. In all cases, the contaminant presence in the effluent water was strongly affected by the substrate nutrient contents, the evaporation rates inside the greenhouse and the extraordinary and punctual necessity of support irrigation with tap water out of schedule (two weekends), due to the temperature rise towards the end of the trial. Overall, organic matter in the influent wastewater (in the form of COD) was easily attenuated (mean attenuation among genotypes of 93.4 %), followed by the EC (79.9 %) and the TN (57 %), which were more affected by the aforementioned factors (Table 2).

Regarding nitrogen transformations and treatment, while  $\text{NH}_4^+$  and organic nitrogen (amines, amides, amino acids, and other vegetal-derived molecules from yeast) are the primary nitrogen forms in influent wastewater,  $\text{NO}_3^-$  predominates in the effluent from both the experimental and control pots. This shift suggests that the elevated nitrogen levels in the effluent from the control pots originate from substrate leaching, as there are no nitrogen sources in the influent (tap water). Consequently, the higher  $\text{NO}_3^-$  levels in the effluent from wastewater-irrigated pots likely result from the mineralization and subsequent nitrification of organic N and  $\text{NH}_4^+$  in the wastewater, coupled with leaching from the soil, thereby affecting attenuation percentages. This hypothesis is enforced when observing the overall trends of nitrogen contents in both effluents over time, where TN and  $\text{NO}_3^-$  concentrations generally decline as nitrogen becomes depleted from the substrate (SM, Fig-S7). Nevertheless, there was an anomaly towards the end of the experiment, when temperatures began to rise (June 2021). The abrupt increase in temperatures and its consequent increase in the water needs of the plants, made necessary an extraordinary irrigation event with clean tap water during the weekend. As a result, TN values in the effluent from wastewater-irrigated pots show a sudden increase during this event, most likely due to the removal of previously adsorbed nitrogen in the substrate (leaching effects).

In a similar way,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ , and  $\text{K}^+$  values also present this sudden rise during this extraordinary event, in contrast to their general decreasing trend throughout the experiment. This, combined with the observed  $\text{Na}^+$  trend, suggests the occurrence of cation exchange processes during the trial. Specifically,  $\text{Na}^+$  from wastewater may replace more beneficial ions in the substrate (i.e.,  $\text{K}^+$ ,  $\text{Mg}^{2+}$ , and  $\text{Ca}^{2+}$ , from least to most beneficial) (Arienzo et al., 2009; Smith et al., 2015). This cation exchange, in addition to the well-known effects on soil hydraulics (Halliwell et al., 2001), could lead to a detriment in the removal of other parameters, such as phosphorus, as the presence of  $\text{Ca}^{2+}$  enhances its precipitation as calcium phosphates in calcareous soils (Bouwer, 1974; Duchaufour, 1984; Meffe et al., 2016).

EC data in the effluent from control pots exhibit a similar trend to TN, with values declining over time due to ion depletion from substrate leaching, albeit with a gentler slope. In contrast, trends in effluent from wastewater-irrigated pots differ (SM, Fig-S7). Initially comparable to control pots, EC values in these pots increase towards the trial's end, particularly after the additional irrigation event with tap water, indicating increased leaching of previously adsorbed ions from the substrate. The observation of higher  $\text{Na}^+$  and  $\text{Cl}^-$  values, these being the main ions present in wastewater, confirms this tendency. While significant attenuation is initially observed in effluent compared to influent (especially for  $\text{Na}^+$ ), values rise notably towards the trial's conclusion as the soil's sorption capacity is exceeded and support irrigation intensifies leaching.

When assessing attenuation efficiencies (Table 2), there must be considered that, besides the aforementioned effect of contaminant leaching in the values, effluent from each pot represents the accumulation of multiple irrigation events throughout the week. Consequently, evaporation and water consumption by the plants will likely enrich the effluent, particularly towards the trial's end when temperatures rise and water availability for sampling decreases. This hypothesis is supported by the fact that effluent from those

**Table 2**

TN, COD and EC attenuation percentages for each tested genotype from the beginning until the end of the experiment (T = 4 months) in the pots trial.

Genotype		TN attenuation (%)	COD attenuation (%)	EC attenuation (%)
Poplar hybrids	'2000 Verde'	72.4 ± 18.6 (55.3 – 90.7)	93.5 ± 5.63 (83.4 – 100)	80.0 ± 13.7 (62.8 – 94.1)
	'AF34'	39.5 ± 32.7 (0.74 – 82.3)	91.6 ± 9.83 (80.82 – 100)	74.0 ± 18.7 (47.0 – 91.7)
	'I-214'	57.5 ± 36.5 (4.76 – 96.6)	98.3 ± 5.25 (90.16 – 100)	79.8 ± 20.3 (49.9 – 99.9)
	'MC'	51.2 ± 30.5 (12.9 – 84.8)	93.3 ± 6.71 (82.6 – 100)	76.6 ± 18.4 (49.6 – 93.4)
	'PO-10-10-20'	62.7 ± 27.5 (24.1 – 100)	90.3 ± 6.95 (79.73 – 100)	83.4 ± 9.1 (66.2 – 90.3)
<i>Populus alba</i>				
<i>Salix</i> spp.	'Levante'	60.7 ± 21.1 (25.0 – 89.9)	94.4 ± 7.87 (78.19 – 100)	80.2 ± 16.0 (58.0 – 94.3)
	<i>S. atrocinerea</i>	57.2 ± 27.2 (12.9 – 94.7)	92.5 ± 4.34 (84.23 – 100)	85.1 ± 11.9 (66.4 – 96.2)
Mean		57.30 ± 28.16	93.40 ± 6.90	79.87 ± 15.28

Values shown are the means calculated ± standard deviation, using the weekly % attenuation. Minimum and maximum values in brackets.

genotypes with greater water availability for sampling (those less affected by evaporation and those with poorer growth rates and, therefore, less water necessities) presented lower pollutant concentration, suggesting less influence from plant uptake and evaporation.

### 3.2.3. Field plantation

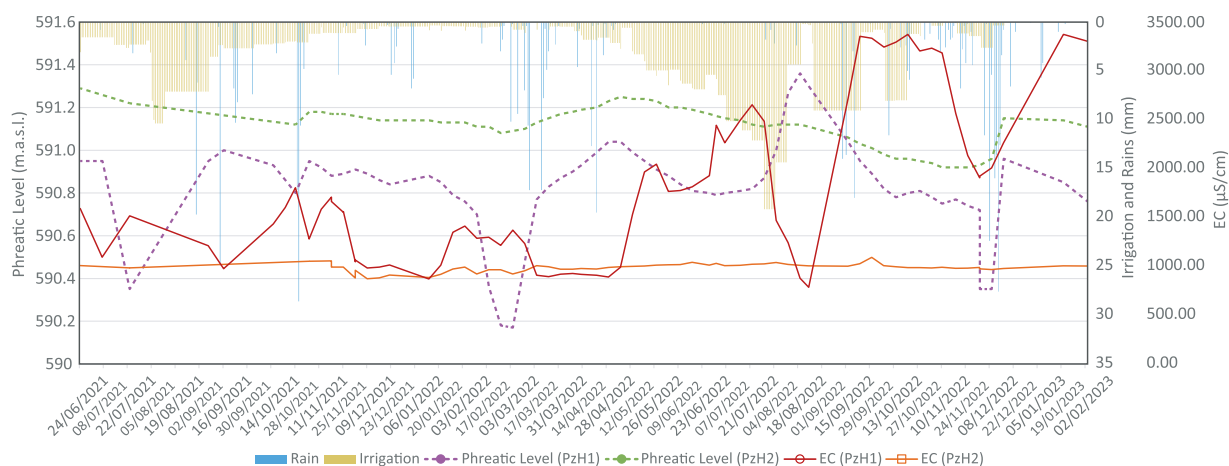
Regarding the VF performance in the field, a different attenuation capacity between the two distinct periods considered can be observed, thus resulting in leaching problems for several parameters during the second one (May 2022 – Dec 2022), and mirroring the same processes observed in the planted pots trials (Figs. 3–4). The performed ANOVA analyses and Duncan's mean separation tests confirm this statistically significant distinction between periods.

Overall, the VF demonstrated average removal percentages of > 99 % for TP, > 98 % for TOC and COD, > 88 % for Na<sup>+</sup> and K<sup>+</sup>, > 87 % for TSS, > 86 % for EC and > 83 % for Cl<sup>-</sup>. For TN, the mean attenuation capacity during the first period was > 75 %, but this significantly decreased during the second period of operation. These removal percentages are very much in consonance with the average values that can be observed in literature when testing VF in different scenarios (Table 3) (Pradana et al., 2021). Also, it is noteworthy that these attenuation rates were, in general, comparable to those achieved by the brewery's conventional WWTP, if not higher (except for the TN), as shown in Table 3. This means that the VF was able to effectively treat the majority of tested parameters as well as the rest of the WWTP process, while also attenuating EC, Na<sup>+</sup> and Cl<sup>-</sup> concentrations (parameters that are not treated by the WWTP), producing biomass and enhancing landscape properties in the site, all with a comparatively much lower maintenance cost (Satya et al., 2025). Moreover, the costs associated with the cutting of the plantation at the end of the cycle (typically the most expensive activity) can also be amortized by using the woodchips as a soil amendment, among other uses (Barbero et al., 2024; Huidobro-López et al., 2023; Meffe et al., 2016).

Time variations in water levels for both piezometers, and their relation to pollutant concentrations in the downstream piezometer (Fig. 3), indicate that water level fluctuations were similar in both piezometers, though more pronounced in the downstream one. This was most likely due to its proximity to the discharge zone of the aquifer, making it more sensitive to general variations in groundwater. The fact that both follow the same trends suggests that variations in the downstream piezometer are primarily influenced by regional groundwater dynamics rather than VF operation, although the latter can accentuate them or interfere, depending on water input. This hypothesis is supported by the inverse correlation that can be observed between water level and pollutant concentration in the downstream piezometer, particularly during the second period (Fig. 3), suggesting that rises in water level respond to a greater input of regional groundwater that dilutes the pollutants infiltrating through the vadose zone. Nevertheless, there were times, especially during the first period of operation (May 2021 – April 2022), when concentration levels in both piezometers were similar. This represents the lowest contaminant concentration that can be achieved in the downstream piezometer, matching the baseline concentration in the local groundwater, consistently shown by the upstream piezometer (Figs. 3–4). Moreover, this base level can be relatively high (e.g., it is of approximately 1000 µS/cm in the case of EC) and thus this must be considered when calculating attenuation percentages and evaluate the treatment performance.

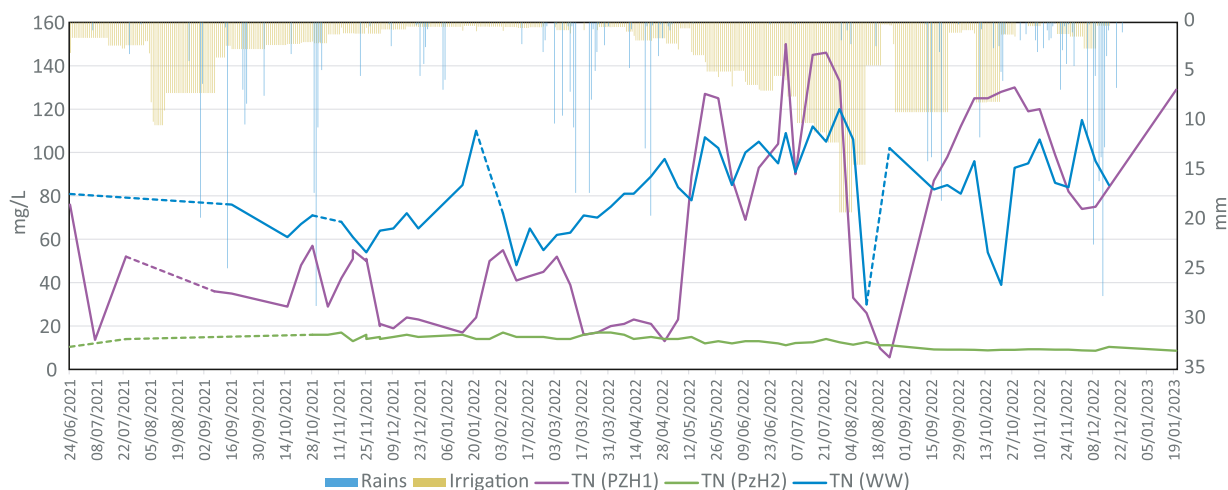
Taking this into account, and considering the values shown by the upstream piezometer as the base level of the aquifer, attenuation percentages can be calculated as in the planted pots trial (Eq. 1), giving the results exposed on Table 3. Further information about the attenuation achieved for other parameters can be found in the SM (Tab-S2).

It is also remarkable the role that rains seem to play in relation to the pollutant concentration in the downstream piezometer, this concentration tends to exhibit noticeable increases around two months after the occurrence of important raining events, suggesting that rain event favor leaching of contaminants, mirroring the effects of irrigation with clean tap water in the planted pots trials and affecting the attenuation percentages (Figs. 3–4). However, this delay could be shortened if the main rain event occurred shortly after



**Fig. 3.** Phreatic levels in both piezometers (green dotted, upstream; purple dotted, downstream), compared to their EC concentrations (orange line, upstream; red line, downstream) and the total input water distribution over time, including irrigation (green bars) and rains (blue bars).





**Fig. 4.** Total Nitrogen concentration in the influent wastewater (blue line) and in both piezometers (blue line, upstream; purple line, downstream), compared to the total water input, including irrigation (green bars) and rain events (blue bars).

**Table 3**

Treatment comparison between the VF and the brewery's conventional WWTP for each operation period.

		Parameter						
		TOC	TN	TP	EC	Na	K	Cl
Mean values (mg/L)	WW	207,42 ± 0,21	80,76 ± 20,08	16,11 ± 4,66	5498,77 ± 1232,76	1483,43 ± 336,50	35,56 ± 8,08	505,88 ± 330,71
	VF (P1)	1,33 ± 0,9	35,10 ± 16,08	0,32 ± 0,27	1126,97 ± 285,28	137,92 ± 24,54	6,20 ± 1,22	127,11 ± 47,53
	VF (P2)	7,93 ± 4,83	95,80 ± 39,36	0,23 ± 0,06	2217,88 ± 783,23	290,07 ± 121,76	8,92 ± 2,19	239,16 ± 112,99
	WWTP	3,24	5,10	1,55	4030	1154	29,59	318,39
	VF (Lit)	99,96	75,08	100 *	96,13	93,99	93,05	93,47
Attenuation (Mean %)	VF (P1)	97,09	-5,23	100 *	77,02	83,79	85,26	73,74
	VF (P2)	98,44	93,68	90,38	26,71	22,23	16,79	37,06
	VF (Lit)	88 ± 12 ***	78 ± 18	80 ± 27	-	-	-	-

WW: Wastewater (secondary); VF: Vegetation Filter (downstream piezometer adjusted values); WWTP: Wastewater Treatment Plant (effluent); VF (Lit): Average VF attenuation in the literature

\*Values in downstream piezometer are lower than the aquifer base level (upstream piezometer).

\*\*Data obtained from our recent review on VF performance (Pradana et al., 2021)

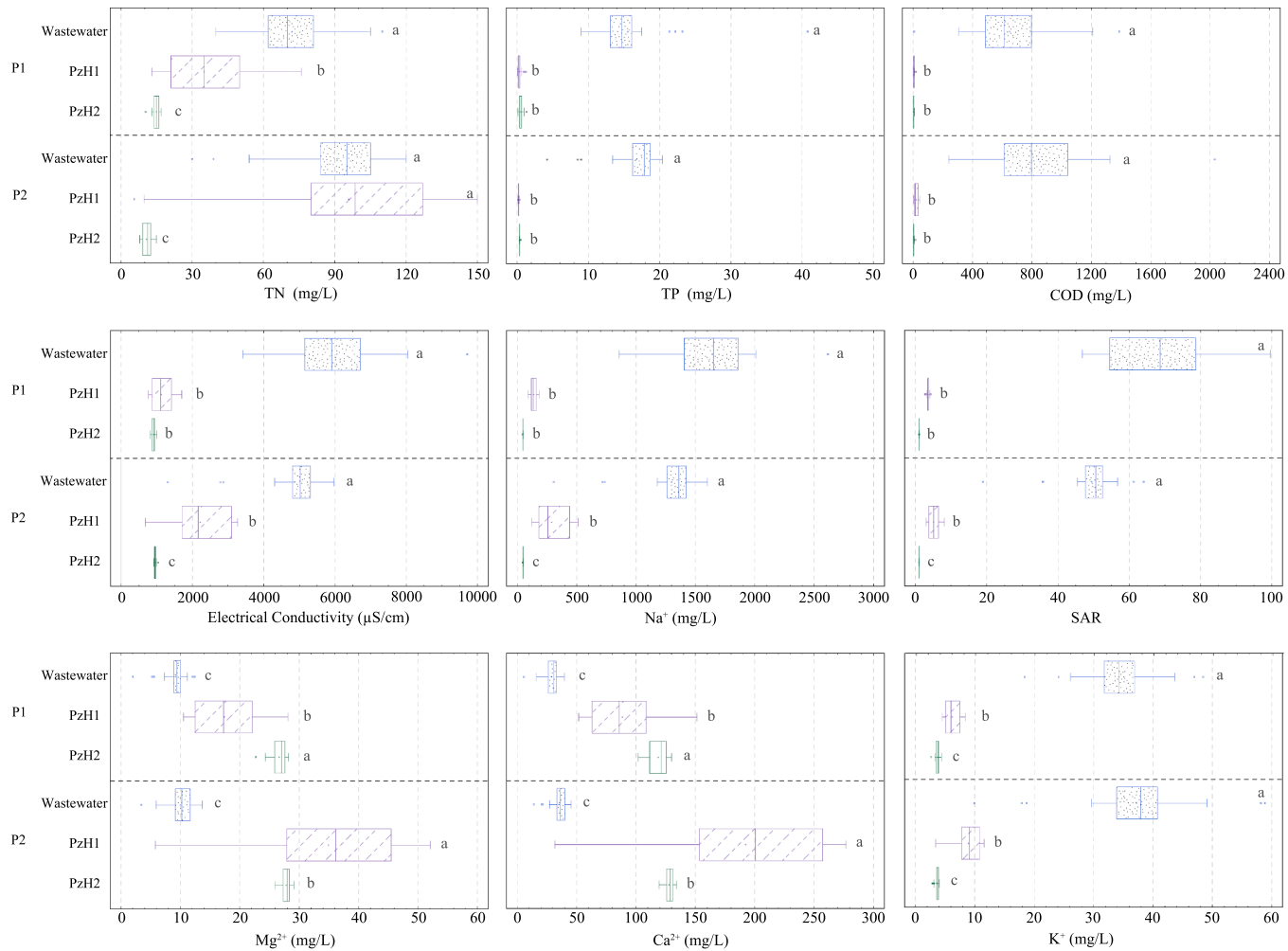
\*\*\*Mean attenuation percentage for Biological Oxygen Demand

Negative values suggest leaching processes occurring.

several minor events, and thus the soil was already wet. During the second period, this effect can even lead to concentrations in the downstream piezometer above those present in the influent wastewater (even adding the base level concentrations in the upstream piezometer), which means that some of this pollution in the downstream piezometer must come from the leachate of contaminants that had been previously retained to the soil (Fig. 4).

The ANOVA analyses and Duncan's mean separation tests performed on the data found statistically significant differences between irrigation wastewater and groundwater in all cases, regardless of the period nor the pollutant (Fig. 5; SM, Tab-S3). However, these differences have also been found for some parameters between the groundwater collected from both piezometers, indicating that the VF operation has an impact in the groundwater from the downstream borehole.

TP (and consequently  $\text{PO}_4^{3-}$ ), COD, and TSS are the only parameters in which no significant differences between both piezometers have been observed. This indicates that the VF was able to eliminate the organic matter, the suspended solids, and the TP present in the wastewater, with only background levels detected in the groundwater. This is consistent with several previous studies, in which these parameters were easily attenuated using VFs (Cavanagh et al., 2011; Curneen and Gill, 2016; Fillion et al., 2009; Pradana et al., 2021). P is easily removed from wastewater used for irrigation of a VF mainly via precipitation in the form of calcium phosphates, sorption to the negatively charged clay particles in the soil and plant uptake of orthophosphate species present in wastewater (Barbero et al., 2024; Duan et al., 2015; Duchaufour, 1984; Pradana et al., 2021). On the other hand, organic matter is mainly removed from wastewater via phytoabsorption and biodegradation by microbiota during their metabolic activity (Barbero et al., 2024; Mant et al., 2003; Pradana et al., 2021). Note that TSS values are higher in the first period than in the second due to the lack of stabilization of the borehole after



**Fig. 5.** Main parameters values in influent wastewater and in groundwater in the field (upstream and downstream of the VF). Different letters correspond to different mean groups in the Duncan's mean separation tests. P1, Period 1 (May 2021 – Apr 2022); P2, Period 2 (May 2022 – Dec 2022); PzH1, downstream piezometer; PzH2, upstream piezometer.

drilling. Nevertheless, these values are still statistically lower than those present in wastewater. EC,  $\text{Na}^+$ , and SAR exhibit similar trends to each other, given that  $\text{Na}^+$  is the predominant ion in the wastewater, influencing EC and SAR values. Statistically significant differences were found between wastewater and groundwater for these parameters in both periods, while differences between piezometers were only found during the second period. This suggests that the VF attenuated these parameters during the first period, but some leaching occurred during the second period.  $\text{Cl}^-$  values followed a similar trend, but with groundwater downstream of the VF showing no significant differences to the wastewater during the second period, indicating even higher leaching. Additionally,  $\text{K}^+$  values also show significant differences between the three sampling points for both periods, indicating an affection to the groundwater. In all cases, even when leaching occurring, the VF managed to achieve high attenuation percentages.

A totally different distribution can be observed for the divalent ions  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{SO}_4^{2-}$ . These parameters were consistently higher in groundwater than influent wastewater. This trend is enforced during the second period, in which the piezometer downstream of the VF shows significantly higher values than the upstream one. This effect suggests that the higher amounts of these parameters in groundwater, especially when affected by the VF operation, are a result of leaching from the soil, most likely due to cation exchange processes during wastewater treatment and percolation. As already stated, this can lead to a detriment in soil hydraulic properties, as the most harmful cations ( $\text{Na}^+$  and, to a lesser extent,  $\text{K}^+$ ) is replacing more beneficial others ( $\text{Mg}^{2+}$  and, especially  $\text{Ca}^{2+}$ ) (Fraps and Fudge, 1938; Smith et al., 2015).

Finally, significant differences in TN were found among all three sampling points during the first period, while no differences were found between influent wastewater and downstream groundwater during the second period. This suggests ongoing N leaching during the VF operation, with a marked increase in the second period, with groundwater values even exceeding those in wastewater (Fig. 5).

Again, these findings align with the literature, as leaching of N in the form of  $\text{NO}_3^-$  into aquifers is a common in VFs assessments (Pradana et al., 2021), and its attenuation capacity can be limited by several factors, like soil drainage capacity, microbial activity, redox conditions or extreme meteorological events (Dimitriou and Aronsson, 2011; Duan et al., 2015; Mantovi et al., 2006; Paranychianakis et al., 2006; Watzinger et al., 2006). Among these factors, the most relevant one is microbial activity. Promoting microbial activity and, therefore, biodegradation, nitrification-denitrification cycles and plant root development, maximizes N removal from wastewater (Barbero et al., 2024; Pradana et al., 2021). Indeed, if wetting-drying cycles in soil are not optimized, the nitrification-denitrification process is hindered, and N leaching tends to occur (Barbero et al., 2024; Li et al., 2012). This microbial biodegradation is especially vital during cold seasons in which plant activity is minimal (Duan et al., 2010; Pradana et al., 2021).

When projecting wastewater and groundwater from both piezometers onto the Piper water characterization diagram and in the USSL diagram for irrigation water adequacy, we can validate some of the aforementioned observations, in both cases especially related to the salinity (and especially sodium) hazard. Graphical representation of wastewater and groundwater samples on these diagrams are helpful when trying to assess tendencies in treatment efficiency over time, focusing on groundwater evolution downstream of the VF (PZH1).

Thus, groundwater in PzH1 appears to be a mixture, primarily from the sodium bicarbonate-type wastewater (notably more alkaline in the second period) and the calcium bicarbonate-type groundwater in the area. This is evident in a Piper diagram, where PzH1 points clearly represent mixtures of brewery wastewater and PzH2 groundwater (SM, Fig-S3), and suggests incomplete treatment with subsequent leaching of  $\text{Na}^+$ . Notably, for both periods, several VF-influenced groundwater samples exhibited higher concentrations of  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$  than the influent wastewater, suggesting leaching from the soil in this industrial area.

During the first period, VF operation increased SAR in the groundwater compared to the upstream samples. However, groundwater downstream of the VF was still within the same diagram field as the upstream groundwater (C3-S1) in the USSL diagram for irrigation water adequacy (SM, Fig-S4). In the second period, groundwater downstream of the VF exhibited rising SAR and EC values, shifting the points into the C3-S2 and C4-S2 fields. This confirms the impact on groundwater, specifically in terms of  $\text{Na}^+$  leaching (among other ions). Monitoring these changes in groundwater composition over time would help identify and prevent environmental and health risks.

To our judgement, there are several factors that can explain why the VF failed to attenuate some contaminants during the second period.

As stated above, the high amounts of  $\text{Na}^+$  present in the wastewater are especially concerning, as the application of  $\text{Na}^+$ -rich water to the soil leads to its detriment, which ultimately affects the overall performance of the VF (Arienzo et al., 2009; Fan et al., 2025; Halliwell et al., 2001; Hillel and Hatfield, 2004; Smith et al., 2015). The main concerns in terms of VF performance in relation to the addition of  $\text{Na}^+$  to the soil are i) soil dispersion and swelling; causing permeability losses; ii) removal of more beneficial cations via cation exchange; and iii) the increase in soil alkalinity. Moreover, this high salinity and  $\text{Na}^+$  amounts can also lead to a phytotoxicity effect (He et al., 2023; US Salinity Laboratory Staff et al., 1954), and a decrease in the hydraulic conductivity of the soil and the subsequent waterlogging can enhance these effects, especially when using an ion-rich and N-rich solution, as in this case (Goyal and Huffaker, 2015). All these detrimental effects are discussed in Section 3.3.

The effects of an intense and sustained drought also must be considered. Indeed, during the second period of the VF operation (i.e. 2022), a drought of such characteristics was present in the euro-Mediterranean region, caused by a persistent anticyclonic anomaly over western Europe. This drought was the most intense since the 19th Century, based on the atmospheric evaporative demand (Bakke et al., 2023; Faranda et al., 2023; Garrido-Perez et al., 2024). This kind of extreme event may have contributed to the detriment of the VF performance, as it must have played a key role in the enhanced mortality rate of the plantation during the second period, like in other forestry scenarios (Allen et al., 2015; Williams et al., 2013). Moreover, high evaporation rates and water uptake by the plants lead to an increase in the concentration of ions in the infiltration water (specifically  $\text{Na}^+$ ), being these concentrations typically 2–3 times higher than in the irrigation water (US Salinity Laboratory Staff et al., 1954), and thus amplifying the sodium phytotoxicity effects. In this case, this problem is even more pressing considering that the study area is in a Mediterranean area, with marked

seasonal temperature and humidity changes (Marcos-Garcia et al., 2017; Pool et al., 2022). The intense nature of rain events following long dry periods with high evapotranspiration rates leads to severe leaching events, a phenomenon expected to worsen over time (Moutahir et al., 2017; Pulido-Velazquez et al., 2015). Indeed, Pool et al. (2022) already found that nitrogen leaching was enhanced after abnormally dry periods, especially using drip irrigation (as is our case) in mediterranean climates.

### 3.3. Impacts on the soil

Textural analyses of the samples collected at the end of the trial, along with their comparison to the initial results, are presented in the SM (Fig-S8). Also, chemical characterization of the same samples is presented in Table 4.

The textural results reveal the presence of two distinct populations: one falls within the clayey loamy category and the other being within the sandy loamy field, in contrast to the pre-VF sample compound, which suggested a general loamy texture, this being the combination of the 2 two observed populations. Soils exhibit a coarser texture towards the upstream sector of the VF (W), while the E sector of the plantation is richer in clay, where more waterlogging was observed. Nevertheless, there is not a clear frontline between the two, and some textural patchwork is present. This is the clearest signal of the high spatial variability present in the soil, most likely derived from the industrial and landfill-like nature of the area for several decades. Furthermore, this textural irregularity also influences the rest of the variables, being the CEC the clearest one, and corroborating that this parameter mostly depends on the texture of the soil, rising with the presence of clay minerals ( $R^2 = 0.915$ ) and the absence of coarse sand and gravel ( $R^2 = 0.939$ ).

Regarding chemical characterization, the VF operation has increased the already high alkalinity of the soil in the site, reaching values over 9, due to the high  $\text{Na}^+$  present in the wastewater. This is consistent with the findings by other authors when applying wastewater (and specifically brewery wastewater) to the soil (Gorfie et al., 2022; Khatun et al., 2019). Indeed, the  $\text{Na}^+$  levels inside the VF area have also been highly increased during the VF operation, along with the EC and  $\text{K}^+$  values, while  $\text{Ca}^{2+}$  levels have been decreased. This, again, supports the insight that cation exchange processes are present, while the total amount of salts in the soil has increased.

The risks of applying  $\text{Na}^+$ -rich water to soil have already been stated. The high  $\text{Na}^+$  concentration interacts with clay particles, causing soil dispersion and swelling, which negatively impacts permeability, redox conditions, the rhizosphere, and overall performance (Fan et al., 2025; Halliwell et al., 2001; Toze, 2006). Therefore, while soil disturbance after plant establishment is generally discouraged to preserve structure (Pradana et al., 2021), in this scenario, regular shallow grading may be beneficial to mitigate high sodicity, break up saline layers, and improve soil structure (Fernández et al., 2004). Moreover, adding organic and inorganic amendments may be crucial for long-term soil health. Organic materials like woodchips or biochar can improve porosity, hydraulic properties, microbial activity, and water retention in sandy areas, while  $\text{Ca}^{2+}$ -rich substances, such as gypsum or calcium lactate, could help ameliorate soil sodification (Clocchiatti et al., 2023; Fan et al., 2025; Gorfie et al., 2022; Huidobro-López et al., 2023; Rashmi et al., 2024).

These considerations highlight the critical need for rigorous control and management of the VF system, as emphasized by Pradana et al. (2021), particularly when compared to other urban wastewater scenarios. Minimizing  $\text{Na}^+$  sources during the previous stages of the brewery's wastewater treatment (i.e., the use of caustic soda, NaOH, during pH stabilization) could significantly improve VF efficiency, whether as an alternative treatment or a supplementary stage to conventional systems. This approach would also prevent soil sodification, eliminating the need for gypsum and reducing the risk of  $\text{SO}_4$  into the aquifer.

### 3.4. Aquifer recharge

The results of the monthly water balance exhibit that irrigation rates did not meet the areas ET, as originally intended (SM, Fig-S9). This is most pronounced during the summer months of both periods of operation, and mostly due to the adjustments made based on the observed waterlogging caused by the infiltration problems occurring in the plantation, forcing us to stop the irrigation before meeting

**Table 4**

Soil samples characterization at the field site – comparison between before and after the VF operation.

Parameter	Mean (2020)	Mean 2023 (VF)	Mean 2023 (non-VF)
pH	8.48	$9.1 \pm 0.63$	$8.13 \pm 0.07$
EC ( $\mu\text{S}/\text{cm}$ )	172	$419.7 \pm 261.4$	$95.9 \pm 4.0$
Clay (%)	22.4	$29.2 \pm 12.5$	$23.5 \pm 12.4$
Lime (%)	31.5	$24.2 \pm 0.7$	$24.6 \pm 3.7$
Sand (%)	46.2	$46.6 \pm 13$	$51.9 \pm 16.1$
TKN (mg/g)	1.29	$1.7 \pm 1.5$	$1.4 \pm 0.2$
Assimilable P (mg/g)	64.8	$49.7 \pm 42.3$	$33 \pm 5.9$
$\text{CaCO}_3$ (g/kg)	42.1	$30.6 \pm 22.4$	$8 \pm 6.2$
$\text{Na}^+$ (mg/kg)	93.8	$1469 \pm 1117.8$	$103.3 \pm 13.8$
$\text{K}^+$ (mg/kg)	258	$362.3 \pm 171.3$	$186 \pm 11.3$
$\text{Ca}^{2+}$ (mg/kg)	3665.2	$2397.3 \pm 1584.5$	$3128.8 \pm 2363.9$
$\text{Mg}^{2+}$ (mg/kg)	539	$641 \pm 321.4$	$386.5 \pm 293.5$
CEC (cmol/kg)	19.8	$21.2 \pm 5.2$	$16.6 \pm 8.6$
Organic Matter (%)	2.65	$2.6 \pm 1.7$	$2.1 \pm 0.1$

EC, Electric conductivity; TKN, total Kjeldahl nitrogen; CEC, Cation Exchange Capacity.

the ET values to avoid further problems. These waterlogging events were especially persistent in the east sector of the VF, where the soil had a finer texture and thus the already stated effects of the interaction between the clays in the soil and the  $\text{Na}^+$  in WW were present. Moreover, mechanical problems involving the pumping system were also present during the summer of 2021. Being the ET in the area higher than the water input, neither waterlogging events in the plantation nor any performance failures in the treatment were caused by an excess in irrigation loads, but because of the already stated effect of  $\text{Na}^+$  interaction with the clay particles in the soil negatively affecting its hydraulic properties.

However, there are several times in which the total water input has exceeded the total expected evapotranspiration, which must have resulted in its infiltration towards the aquifer. The months in which percolation towards groundwater is occurring are those corresponding to the autumn and winter seasons during both periods of the VF's operation, as the ET is significantly lower, the humidity is higher, and the weather is rainier. Adding the amounts of water surpassing the ET for all these months, a total of  $482.1 \text{ m}^3$  of the total water applied to the VF via both irrigation and rain have been used to effectively recharge the underlying aquifer.

#### 4. Conclusions

The findings obtained from the different trials carried out in this work provide some valuable insights concerning the capacity of VFs as a phytoremediation tool for the treatment of secondary wastewater from the brewery industry. The 'Lab-to-field' approach presented here has already proven to be of particular interest for testing the suitability of different plant materials for use in VFs. Although it was not equally decisive in terms of attenuation capacity, it was possible to verify that the processes under controlled conditions faithfully reflected those observed later at field scale, thus validating the experimental procedure and allowing it to be used as a quick and simple first contact in new scenarios.

Overall, the VF has exhibited mixed results concerning treatment capacity but has proven to be able to achieve good attenuation percentages in all the parameters studied. These percentages were especially high for TP, organic matter and TSS, and, excluding TN, they were always comparable or even higher than those achieved by the WWTP of the brewery. In this sense, the overall performance in terms of EC,  $\text{Cl}^-$ ,  $\text{Na}^+$  and SAR attenuation is of special importance, as the treatment of these parameters is overlooked when evaluating conventional treatment systems. Nevertheless, it would be of very high interest to minimize the use of these ions during the first stages of the brewery's WWTP procedure when possible (i.e., using salts with less deleterious cations for soil properties and plant development, such as  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ , or even  $\text{K}^+$ , as pH neutralizers), given their detrimental effects on the soil and, therefore, on the system performance during the second period of activity.

One way or another, it would also be advisable to ameliorate the effects of high salinity and sodicity via cultural practices, such as grading, and the application of soil amendments, using both organic and inorganic substances. Moreover, the use of these kinds of organic amendments would help reduce the structural and textural variability of the soil, given its industrial and landfill-like nature, thus palliating its detrimental effects and enhancing overall performance.

#### CRediT authorship contribution statement

**Hortensia Sixto:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **Nerea Oliveira:** Visualization, Software, Methodology. **Irene De Bustamante:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Isabel González:** Resources, Methodology. **Borja Daniel González-González:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization. **Raúl Jerónimo Pradana Yuste:** Writing – original draft, Visualization, Validation, Software, Resources, Methodology, Investigation, Formal analysis, Data curation, Conceptualization.

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#### Declaration of Competing Interest

The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

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#### Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.eti.2025.104227](https://doi.org/10.1016/j.eti.2025.104227).

## Data availability

Data will be made available on request.

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