

UNIVERSITAT DE BARCELONA

Subsurface flow constructed wetlands for the treatment of wastewater from different sources. Design and operation

Antonina Torrens Armengol

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SUBSURFACE FLOW CONSTRUCTED WETLANDS FOR THE TREATMENT OF WASTEWATER FROM DIFFERENT SOURCES. DESIGN AND OPERATION

Antonina Torrens Armengol

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SUBSURFACE FLOW CONSTRUCTED WETLANDS FOR THE TREATMENT OF WASTEWATER FROM DIFFERENT SOURCES. DESIGN AND OPERATION

Memòria presentada per Antonina Torrens Armengol per optar al títol de doctor per la Universitat de Barcelona

> Tutor i director Dr. Miquel Salgot de Marçay

Doctoranda Antonina Torrens Armengol

2015

Als meus pares

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ABBREVIATIONS AND ACRONYMS

%VS	Percentage of Volatile Solids
ACA	Agència Catalana de l'Aigua
bdl	below detection limit
BOD ₅	5 day Biochemical Oxygen Demand
C/N	Carbon/Nitrogen
cfu	colony-forming unit
Chl-a	Chlorophyll a
COD	Chemical Oxygen Demand
CU	Coefficient of Uniformity
CW	Constructed Wetland
d ₁₀	Mesh diameter allowing 10% of the sand mass to pass through (mm)
d ₆₀	Mesh diameter allowing 60% of the sand mass to pass through (mm)
dCOD	dissolved Chemical Oxygen Demand
dl	detection limit
DM	Dry Matter
DO	Dissolved Oxygen
DTD	Detention Time Distribution
E(t)	Detention Time Distribution function
E. coli	Escherichia coli
EC	Electrical Conductivity
EC	European Community
EH	Oxidation-reduction potential
FC	Fecal Coliforms
FNU	Formazine Nephelometric Unit
FWSCW	Free Water Surface Constructed Wetland
HCW	Hybrid Constructed Wetland
HDPE	High Dendity Polyethylene
HFCW	Horizontal Flow Constructed Wetland
HL	Hydraulic Load
HRT	Hydraulic Retention Time
IP	Infiltration-Percolation
IR	Infiltration Rate
IRSTEA	Institut national de recherche en sciences et technologies pour l'environnement
	et l'agriculture (former CEMAGREF)
Log cfu	Logaritmic colony-forming unit
Log pfu	Logaritrimic plaque-forming unit
m/s	meters/second
max	maximum value

min	minimum value
mIP	modified Infiltration-Percolation
$N-NH_4^+$	Ammonium
N-NO ₃ ⁻	Nitrates
NTU	Nefelometric Turbidity Unit
OLD	Organic Loading Rate
pCOD	particulate Chemical Oxygen Demand
PE	Person Equivalent
pfu	plaque forming unit
P-PO4 ³⁻	Phosphates
PVC	Polyvinyl Chloride
RD	Royal Decree (Spanish Regulation)
SD	Standard Deviation
SDRB	Sludge Dewatering Reed Bed
SS	Suspended Solids
S-SO4 ²⁻	Sulphates
ST	Settling Tank
tHRT	theoretical Hydraulic Retention Time
TKN	Total Kjeldahl Nitrogen
TN	Total Nitrogen
тос	Total Organic Carbon
TP	Total Phosphorus
TS	Total Solids
UB	Universtitat de Barcelona
ULog	Logaritmic Units
UN	United Nations
US EPA	United States Environmental Protection Agency
VS	Volatile Solids
WHO	World Health Organization
WWTP	Wastewater Treatment Plant

ABSTRACT

Natural technologies for wastewater treatment offer certain advantages such as simple construction, easy operation and cost-effective maintenance. Constructed wetlands, mainly with subsurface flow, are some of the most common natural technologies used to treat domestic and municipal wastewater, for secondary or tertiary treatment. Recently, constructed wetlands have been also applied for the treatment of wastewater from different activities (agriculture, industrial, landfill leachate, etc.). The specific characteristics of this wastewater (new pollutants, extreme concentrations, low biodegradability, high toxicity) is a challenge for the application of subsurface flow constructed wetlands, and further research is necessary to optimize their design and operation.

The aim of this thesis is to examine the viability of the subsurface constructed wetlands for the treatment of wastewater derived from three different sources (treatment ponds, pig farms and car wash facilities), and to evaluate the influence of design (size, type and depth of media, presence of *Phragmites australis*) and operational parameters (hydraulic load, dosing and feeding modes) on treatment efficiency and hydraulic behavior.

The studies were done in the framework of different national and European R+D projects. The viability of constructed wetlands with vertical and/or horizontal subsurface flow to treat the effluent from wastewater treatment pond systems (facultative and tertiary ponds) for discharge or reuse was evaluated in the framework of LILIPUB (LIFE3-ENV-F303, coordinated by IRSTEA-Lyon) and MEDIWAT (1G-MED09-262, coordinated by Water Observatory, Sicilian Region Service) projects. It was done in two municipal wastewater treatment plants, in Aurignac (France) and in Santa Eugènia (Mallorca, Spain). The viability of hybrid subsurface flow constructed wetlands to treat swine slurry from the small farm Can Corominas located in Viver i Serrateix (Barcelona, Spain) for land application or discharge was evaluated in the framework of CTM2010-19197 and MEDIWAT projects. Finally, the viability of vertical and horizontal flow constructed wetlands to treat the effluent from car wash facilities for recycling at a car wash station located in Montfullà (Girona, Spain), using an infiltration-percolation system as reference, was evaluated in the framework of MinAqua project (LIFE11-ENV-569, coordinated by Fundació Ramón Noguera).

The main outcome of the thesis is the viability of the application of different configurations of subsurface flow constructed wetlands to treat the effluents from a wastewater treatment pond, a pig farm, and a car wash facility, once design and operation have been optimized. Pre-treatment is required in some cases due to the specific characteristics of the effluents treated. Subsurface flow constructed wetlands have proved to be a sustainable and efficient technical solution to treat small wastewater flows with special characteristics. Subsurface flow constructed wetlands have shown resilience to load and hydraulic fluctuations, to new pollutants and to variable environmental conditions; being simple to operate and maintain with null or minimum energy requirements and with an added aesthetical value.

Paying special attention to the pond effluent study, it was characterised by a large quantity of algae and a high effluent variability that depends on environmental factors (temperature and solar irradiance). The experimental results demonstrated the effectiveness of vertical and horizontal flow constructed wetlands to upgrade pond effluent quality by retaining algae and suspended solids, completing organic matter degradation, and nitrifying the pond effluent in the case of vertical flow and partially removing total nitrogen in the case of horizontal flow. Retention of phosphorus was overall very low. The granulometry laser technique to determine the size and number of particles in the water samples was useful to characterise suspended solids concentration and to study the filtration capacity of subsurface flow constructed wetlands.

The filtering media size (sand or gravel) was the key parameter for algae retention. This was especially important for vertical flow constructed wetlands, where the choice of the sand size was a main parameter to achieve a good filtration, provide enough hydraulic retention time and avoid clogging. The presence of plants did not significantly affect the filter performance, although it was important in terms of maintenance and temperature moderation. The deeper the filter, the better performance for all parameters in vertical flow constructed wetlands, with a significant effect on algae retention. Overall, the increase in hydraulic load reduced removal efficiency in both types of subsurface flow constructed wetlands. Hydraulic retention time in vertical flow sistems was strongly influenced by the fractionation of the daily hydraulic load, and also played an important role in determining the treatment level. The removal of microbial indicators depended mainly on the water retention time in the filter, which in turn depended on the media granulometry and hydraulic load (for both horizontal and vertical flow constructed wetlands), and on the depth of the filter and the dose volume per batch (for vertical flow constructed wetlands, which were not limited by low temperatures). Bacterial indicators were removed at a higher rate than viral ones. Somatic coliphages in turn were removed at higher rates than F-specific bacteriophages.

On the other hand, swine slurry presented high concentrations of suspended solids, organic matter, nitrogen and phosphorous, with high variability depending on the farm management and storage conditions of the slurry. The hybrid configuration, which combines vertical and horizontal flow constructed wetlands, had a dual function for simultaneous solid-liquid separation and biological treatment. Removal of organic matter and suspended solids was very high, while the overall nitrogen load removal was 63%, due to the combined nitrification/denitrification processes. Vertical flow constructed wetlands were operated intermittently and with sequential feeding, achieving good hydraulic performance with no clogging problems, despite high pollutant loads. The number of feeding/resting days to be applied in vertical filters depends on the organic load, hydraulic load, fractionation of the hydraulic load and temperature. Some of the pollutants were retained and mineralized in the surface deposit layer, increasing around 20 cm each year. This organic biosolid layer improved filtration efficiency. The high ammonia contents interfered with the growth of *Phragmites australis*, while the high cncentration of suspended solids and organic matter also limited the type of subsurface flow constructed wetland to be implemented.

Finally, car wash effluent had a high concentration of inorganic suspended solids, very variable concentrations of E. coli and organic matter, low concentrations of nutrients, and the presence of hydrocarbons, fats and oils. The studied car wash effluent presented non-ionic surfactants, but at lower concentrations than expected due to the high dilution, and high biodegradability and low dosing of the detergents used in the car wash facility. The three technologies evaluated performed very efficiently with respect to turbidity, organic matter and suspended solids. Nonionic detergents, hydrocarbons, fats and oils were also completely removed, but their concentrations in the influent were already very low. E. coli was removed to acceptable limits for recycling, with concentrations lower than the limits established in the Royal Decree for reuse in Spain. The oil and fats contents of the car wash effluents as well as the inorganic suspended solids made pre-treatment necessary in order to avoid media clogging. This was especially important for the horizontal flow constructed wetland, and for the drip irrigation of the infiltrationpercolation system. The vertical flow constructed wetland performed without any clogging problems throughout the study for all of the applied loads, even without resting periods. Additionally, the low concentrations of nutrients resulted in a slow growth of Phragmites australis.

1. INTRODUCTION

1. INTRODUCTION

1.1. Natural technologies for wastewater treatment

Natural technologies ("extensive technologies", "soft technologies", "non-conventional technologies" or "sustainable technologies") for wastewater treatment use natural, commonly occurring self-treatment processes that take place in soil, water and wetland environments. Soil and vegetation are directly involved in the processes, mainly through the formation of favourable conditions for the development of microorganisms taking part in the treatment process. These technologies are defined in terms of the presence of natural components or complete systems (ecosystems) in the wastewater treatment.

These systems can use unique natural components (e.g. soil) or more complexe systems with various components or entire ecosystems. The processes involved in these technologies in order to eliminate wastewater pollutants tend to be similar to those used in conventional systems (aerobic or anaerobic biological degradation, oxidation and reduction reactions, sedimentation, filtration, etc.) which are combined with other naturally occurring processes of the ecosystems (photosynthesis, microorganism or plant assimilation, etc.) (Vera *et al.*, 2006). The main difference between conventional and natural technologies (Table 1.1.) is that the former tend to occur in energy-accelerated velocity reactors, while in the extensive systems, the processes occur at "natural" speeds (without the addition of artificial energy). These energy savings are compensated for by the need for a larger surface area (thus their name, "extensive"). That is, since they treat the same pollutant load, the natural systems require considerably larger surface areas.

Conventional or intensive systems, frequently used in wastewater treatment in large communities, are not always appropriate for smaller areas. This is mainly due to the energy consumption and complex operation and maintenance of these technologies. Also, intensive systems are not always appropriate for small or seasonal villages, where flows are not continuous and may vary enormously on a daily, weekly or seasonal basis (Tchobanoglous *et al.*, 2003).

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Conventional	Natural
High energy expenditure: electrical energy for	Little or no energy expenditure: Natural energy (sunlight
oxidation and mixing in reactors (higher cost)	and occasionally wind energy)
Advanced technological equipment	Little or no advanced technological equipment. Ground
Advanced technological equipment	movements in construction are vital
Proportionally lower surface area	Require considerable surface area
Short hydraulic retention time	Long hydraulic retention time
Processes may be rapdly modified	Treatment mechanisms have considerable inertia
Complex maintenance and exploitation	Simple maintenance and exploitation
Specialized labour supply	Management should know the processes and be able to
Specialized labour suppry	prevent problems
Technological appearance	Good integration in landscape
Artificial processes (very accelerated systems)	Natural processes at "natural" speeds

Table 1.1. Comparison of conventional and natural technologies

Natural technologies have been established across the globe as an alternative to conventional mechanical wastewater treatment systems in small communities, serving as main secondary treatment systems. The capacity of these technologies to remove pathogen indicators (Torrens *et al.*, 2009b) has also led to their use as tertiary treatment systems in large communities. Ponds, infiltration-percolation (IP) and constructed wetlands (CWs) are often as polishing treatments used subsequent to conventional treatments (e.g. activated sludge) for wastewater reclamation.

Another common application of natural technologies is their use in decentralised sanitation. In many parts of Europe, especially in the high tourism coastal zones, there are numerous isolated houses, groups of homes, camping grounds, hotels, etc. In these cases, centralised wastewater management systems are often impossible or very difficult to implement due to the long sewer networks that are required. Due to the variable wastewater flows and relatively remote locations typical of on-site and decentralized treatment systems, technologies must be robust and capable of operating with minimal maintenance or supervision (Nivala *et al.*, 2013). Nowadays, natural technologies (especially CWs) are more and more frequently used for decentralized applications: for wastewater treatment, for disposal to the environment (usually instead of septic tanks) or for reuse and recycling (for both black and grey water). The use of these technologies has also extended to the treatment of various industrial, agricultural anc cattle effluents since the operators of small industries or farms are not always able to afford conventional effluent treatment systems.

There are a number of natural technologies and classifications based on the most important system component or type of biomass used (Figure 1.1.).



Figure 1.1. Classification of natural technologies

These technologies may also be combined. The association of several natural systems, in series or in parallel, is sometimes implemented in order to adapt the treatment to a specific goal (quality of discharge, special characteristics of the influent such as industrial wastewaters, integration of rainwater, etc.) (Salgot and Torrens, 2008).

Within a specific natural technology, different types of the technique are also combined to achieve specific objectives. These configurations will be explained in each technology section. The most frequently used natural technologies for wastewater treatment are ponds, IP and CWs. A summary of the characteristic of the ponds and IP systems are presented in the subsections below. CWs are described in more detail in section 1.2.

1.1.1. Ponds

Ponds have been used as a widespread method of wastewater purification based on the complex interactions occurring in aquatic stagnant ecosystems. The natural mechanisms occurring are of a physical, chemical and biological nature and permit the elimination, inactivation or transformation of pathogenic microorganisms, organic matter, suspended solids (SS) and nutrients, among others. The most important activities are carried out through the metabolic activity of bacteria and algae, at a limited speed depending on environmental conditions. This is the oldest and most well-known natural wastewater treatment system (Mara and Pearson, 1998).

Physically speaking, ponds are lagoons in which the water to be treated is retained for relatively long periods of time (several days or weeks) and in which there is typically the substitution of organic fecal material with new organic material that is basically in the form of algae. These ponds are created based on the number of person equivalents (PE), of organic matter to be treated or the disinfection to be achieved. In situations with few PE, one single pond is constructed; but in larger installations there may be a series of ponds; finally, several parallel lines are used when possible. The key element for efficiency of these ponds is the hydraulic retention time. Pond systems (also known as Waste Stabilisation Ponds) are typically formed by a variety of types of ponds, classified based on the distribution of dissolved oxygen in the body of water:

- Anaerobic
- Facultative
- Maturation (aerobic)
- Storage

The ponds can be used individually, or linked in a series for improved treatment (Tilley *et al.*, 2014). There are different configurations such that the distribution and sequencing of the ponds is quite diverse, according to the final quality of the effluent that is to be attained and the space that is occupied. In the design of any type of pond, it is necessary to consider the geometry, layout and number of inlets and outlets. The most commonly used shape is the rectangle, with variations in the length-width relationship. An optimal geometry and correct layout of the inlets and outlets as well as the positioning in regards to dominant winds may minimize the existence of short circuits and dead zones, while at the same time increasing the efficiency of the pond treatment (Salgot and Torrens, 2008).

 <u>Anaerobic ponds.</u> Anaerobic ponds are used for the initial treatment of wastewater and therefore, they are designed to receive a very high organic load, meaning that they are virtually free of dissolved oxygen and algae. Their main function is to eliminate solids and organic matter in suspension through sedimentation and subsequent anaerobic digestion. The anaerobic ponds have relatively small surface areas and a typically depth of between 2 and 5 m, with a short hydraulic retention time, between 1 and 6 days.

- Facultative ponds. Facultative ponds are characterized by the presence of aerobic conditions in the upper layer and anaerobic conditions in their bottom. Thus two distinct zones exist: a superficial one, where the stabilization of organic material occurs based on oxidation with dissolved oxygen, and a deep zone, where anaerobic degradation reactions predominate. This type of pond is used to directly treat urban wastewater or to receive effluents treated by anaerobic ponds. Although part of the oxygen is provided by the atmosphere, the majority comes from photosynthetic activity of the numerous algae present in the pond, thanks to its high number and sunlight. Its main function is to eliminate non-settleable organic matter, although it is also to eliminate pathogenic microorganisms and nutrients. Facultative ponds have usually a depth of between 1.5 and 2 m. If the pond performs correctly, it will have a bright green colour due on the presence of algae. Facultative ponds operate based on synergy between microorganisms - mainly bacteria - and algae. These algae consume nutrients and produce oxygen, used by heterotrophic microorganisms to oxidize organic materials, generating nutrients that are used by the algae. Wind is the main source of energy in the mixture of the facultative pond water, although depending upon climatic conditions, differential heating is another cause of mixing. Mixing is a major physical parameter that affects the growth of the algae, given that many algae are not mobile and require the mixing in order to access the area of effective light. Furthermore, during daytime hours, mixing contributes to the distribution of oxygen.
- <u>Maturation ponds.</u> Maturation ponds, also known as polishing ponds, contain dissolved oxygen (often in oversaturation) in virtually all of its volume (usually between 1 and 1.5 m of depth), and always operate following the other purification processes, since in order to maintain their aerobic conditions, they must receive a very low organic load. Their main function is to eliminate pathogenic microorganisms (disinfection). The primary biochemical reactions occurring in these ponds include aerobic oxidation of organic material and photosynthesis. Pathogenic microorganisms are eliminated by the elevated temperatures, pH (basic), light (UV radiation) and concurrence of the micfroorganisms. The algae population differs in comparison to that of the facultative ponds.
<u>Pond reservoirs</u>: they are used for the accumulation of water used for irrigation. These ponds are located at the end of the pond series or after the conventional systems in which wastewater must be reused. They receive low organic loads, are large in size and depth (10-5 m depth) and very high and changing hydraulic retention times (months). The reservoirs present aerobic and anaerobic zones, and upgrade the water quality (mainly pathogens and nutrients).

Today, pond systems continue to be regarded as one of the top methods for wastewater treatment in many parts of the world. In France and Germany (Boutin *et al.*, 2005), facultative ponds are very widely used in small rural communities (generally up to 2000, however larger systems exist in Mediterrean). In the United States, in 1980, one third of all wastewater treatment plants were pond systems, usually serving small-medium urban populations (Mara, 2004). In warmer climates (the Middle East, Africa, Asia and Latin America) ponds are typically used for large populations (up to around 1 million) where land is often available at reasonable cost. Due the high disinfection capacity of these systems, they tend to be used when a high microbiological water quality is required for reuse (mainly agricultural reuse). Pond systems (anaerobic basins) are also used as for cattle raising facilities wastewater (e.g. piggery effluents) both for accumulation and treatment before disposal to the land.

This technology may be combined with others (e.g. maturation or macrophyte ponds as tertiary treatment systems following conventional technologies for wastewater reuse purposes). Sometimes, complementary techniques are applied after ponds in order to upgrade their effluent quality, removing the effluent algae (Torrens et al., 2006a). Methods of algae recovery are also applied to use recovered biomass for energy production. However, most of the harvesting and recovering algae applications do not occur with natural ponds but with High Rate Algal Pond systems (HRAP) (Park and Craggs, 2010). High rate algal ponds (HRAP) form part of an advanced pond system first developed in the 1950s for the treatment of wastewater and nutrient recovery in the form of microalgal biomass. The central concept behind HRAP wastewater treatment is that microalgae photosynthesis provides the necessary oxygen that drives aerobic bacterial degradation of organic compounds, which, in turn, provides the CO_2 required for photosynthesis (Oswald, 1988). Coupled with bacterial breakdown of organic compounds, microalgae are able to directly assimilate soluble organic compounds thus contributing to the chemical oxygen demand removal. The design of HRAPs allows for microalgae to grow profusely which enhances nutrient removal through assimilation into their biomass. This results in combined secondary and partial tertiary wastewater treatment within the HRAP (Sutherland *et al.,* 2015).

The main advantages and drawbacks of pond systems are listed in Table 1.2.

Advantages	Drawbacks			
An energy supply is not necessary if the difference in level is favourable (solar energy and wind as the natural energy)	Considerable ground space required (around 10 m ² /PE)			
Adapts well to large variations in hydraulic load	Final effluent with suspended solids and organic matter from algae			
Operation remains very simple	High evaporation with reduced water flows in summer and possibility of increasing the salinity of the final effluent			
No "hard permanent" constructions, civil	Quality of discharge varies according to season (very			
engineering remains simple	sensitive to low temperatures and low solar radiation)			
Integrates well into the landscape, and absence of noise pollution	Facultative ponds are very sensitive to high strength influents, so that are not usually used for most of industrial wastewaters			
Very good elimination of pathogenic bacteria (particularly in summer)	Controlling the biological balance and purification processes remains limited			
Possibility of reuse of the effluent and the produced biomass (algae)	Risk of odours when disfunctioning (particularly in anaerobic ponds)			
Good removal of nutrients: phosphorus and	Risk of mosquito presence when poor maintenance			
nitrogen in summer	(presence of plants in the banks)			
Sludge is well stabilised except for that which is present at the head of the first basin	Investment costs depend very heavily on the type of substratum. With unstable or sandy land, it is preferable not to consider this technology			

1.1.2. Infiltration-percolation

Infiltration-percolation of wastewater is a treatment process by aerobic biological filtering through a fine granular medium. IP tecniques appeared in the United States during the 1940s, as extensive secondary treatment for small and medium communities. These types of facilities were adopted in other countries with several modifications, especially in France and Israel. In France, the first IP system was installed in 1981 in Port Leucate, where secondary effluent produced during summertime period is infiltered and percolates feeding a phreatic layer (Brissaud and Lesavre, 1993). During the late 1980s, modified Infiltration-percolation (mIP) was

developed in France, with the goal of achieving greater independence from the hydrological context and extending the IP application field. The mIP arrived in Spain in 1991 (Brissaud *et al.*, 2007), and other improvements were established, mainly the application of homogeneity of application. The mIP is usually used as tertiary treatment system for wastewater reaclamation and further reuse.

This is an aerobic process; oxygen is supplied during the passage of water through the system and via gaseous exchanges with the atmosphere. IP is a porous system by definition, acting on the pollutant load mainly through two mechanisms: surface filtration and biological oxidation. These mechanisms allow for three main objectives of purification to be achieved: the almost total elimination of the suspended solids and organic particulates; oxidation of the dissolved organic matter and nitrogen transformation (nitrification); and a major reduction of the number of pathogenic microorganisms.

An IP system should include previous storage and impulsion/delivery systems, a feeding device, one or more sand beds and an effluent evacuation system. The filtering body is typically made up of sand, conveniently calibrated and washed. This material allows for the obtaining of homogenous filtering bdies, necessary for good control of the infiltration. The granulometry of the sand should be sufficiently large so as to ensure the rapid renewal of the gaseous phase, but at the same time, it should be sufficiently fine so as to retain part of the suspended solids and to limit the percolation velocity. Table 1.3 presents a summary of the recommended characteristics for the sand in these systems.

Characteristics of the sands			
Sand types Silica / other sand that is resistant to abrasion an			
	the chemical action of water		
Washing	Yes		
d ₁₀	Between 0.25 mm and 0.40 mm		
CU (d ₆₀ /d ₁₀)	Between 3 and 6		
Percentage of fines	Less than 3 %		

Table 1.3. Recommended characteristics for the sand in the IP systems (modified from Lie	nard
<i>et al</i> ., 2001)	

d₁₀: Mesh diameter allowing 10% of the sand mass to pass through (mm), CU: Coefficient of uniformity: ratio d₆₀/d₁₀, d₆₀: Mesh diameter allowing 60% of the sand mass to pass through (mm)

The depth of the bed depends on the goal of the IP system. If the elimination of the indicators of fecal contamination is not an objective of the installation, a filtering massif

thickness of 70 cm is sufficient. If IP has the goal of the elimination of the pathogenic microorganisms, the thickness of sand shall depend upon the level of disinfection anticipated. In general, a thickness of 1.5 m of sand is recommended. Feeding of the beds is always carried out in an intermittent way. Normally, the water reaches the feeding device from a regulating tank. These tanks store the water and send it to the distribution system via a siphon or pumps that are regulated by height sensors. The simplest of installations may work strictly with gravity, that is, without pumping or electrical installations. The emptying of the storage tank can be done with a siphon or a pendulum. The feeding device of the infiltration units should ensure a uniform distribution of the influent (in order to use the entire available surface area) and the homogeneity of the applied hydraulic loads. Feeding may be carried out through temporary immersion or sprinkling. The most commonly used irrigation system in the infiltration percolation system is via immersion, through perforated pipes. These pipes are distributed so as to permit a homogenous distribution of the influent in the surface of the filtering body. The daily hydraulic load is fed in an intermittent way via batches. Application of water in a sequential way ensures the necessary oxygen so as to provide oxidation mechanisms for the pollutant load. For the proper management of these systems, it is important to have various filters constructed in parallel, in order to give the necessary rest time to each filter to prevent clogging.

The IP systems may also be fed via aspersion (mIP). In these cases, the wastewater is applied via sprinklers. The distribution in this case consists of regularly spaced pipes having nozzles or blades in their lower part, depending on the type of wastewater. Alternating between phases of feeding and resting requires a programed water delivery to the infiltration surface. Like irrigation via immersion, the feeding should be sequential to ensure that the gaseous phase is renewed (Brissaud *et al.*, 2007). The evacuation of the effluent in these systems is carried out via a perforated flexible tube located in the gravel level. Recent innovations of the mIP technique include the use of subsurface drip irrigation in a sand body planted with grass so as this technology can be applied as tertiary treatment in urban environments (e.g. roundabouts). The principal advantages and drawbacks of IP systems are listed in Table 1.4.

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Advantages	Drowbooko
Advantages	Drawbacks
Provides high water quality effluent: high removal of BOD ₅ , COD and SS	Almost exclusive use for urban wastewater
High level of nitrification	Requires great quantities of sand to be available, which could lead to high capital cost if none is available
	nearby
Excellent capacity of disinfection	Requires an effective primary settling
Required surface area is much less than for natural ponds (1-2 m ² PE)	Limited adaptation to hydraulic overloads
Maintenance remains simple but more "demanding " than ponds	Sensitive to freezing
	The risk of clogging must be managed (hence the
Moderate investment costs	importance of the use of a "washed" sand with good
	sizing) and the resting periods respected

Table 1.4. Advantages and drawbacks of IP systems

1.2. Constructed wetlands for wastewater treatment

1.2.1. Definition and background

CWs are engineered systems, designed and constructed to utilise the natural functions of wetland vegetation, soils and microbial populations to treat wastewater pollutants. CWs consists of impermeable basins, which use engineered structures to control flow direction, water retention time and water level (US EPA, 2000). Constructed wetlands are planted with aquatic macrophytes, typical of natural areas and used to treat wastewater from different sources. A variety of terms are used in regards to CWs, including: man-made, engineered, artificial or treatment wetlands.

Initial experiments on the use of wetlands for wastewater treatment were conducted by Seidel in the 1960s (Seidel, 1961) and by Kickuth in the 1970s (Kickuth, 1978). During the early stages of CW development, the application of CWs was used mainly in the treatment of traditional tertiary and secondary domestic/municipal wastewater systems (Kivaisi, 2001). The first CW began operating in 1967 in the Netherlands. Although CWs were initially used for domestic and municipal wastewater, aimed at inexpensive and effective ecological wastewater treatment, the application of CWs has also been significantly extended to other type of wastewaters (e.g. industrial, agricultural, runoff) using different configurations, scales and designs. CW applications are described in

section 1.2.5. Knowledge of CWs for wastewater treatment over the past five decades has been reviewed and summarised by different authors (Reed *et al.*, 1995; Cooper *et al.*, 1996; Kadlec and Knight, 1996; US EPA, 2000; UN Habitat, 2008; Kadlec and Wallace, 2008; Bresciani and Masi, 2013; Stefanakis *et al.*, 2015). Some recent papers reviewing the state of the art of CWs in different fields are listed in Table 1.5.

Торіс	References			
	Vymazal, 2011			
General	Fonder and Headley, 2013			
	Vymazal, 2013			
	Faulwetter <i>et al.,</i> 2009			
Removal mechanisms	Vymazal and Kopfelova, 2009			
	Vymazal, 2013			
Modelling	Langergraber, 2008			
	Vymazal, 2014			
Industrial applications	Wu <i>et al.,</i> 2015			
	Vymazal, 2013			
Wetland vegetation	Shelef, 2013			
Developments, challenges and status in	Zhang <i>et al.</i> , 2009			
different countries	Wu <i>et al.</i> , 2015			

Table 1.5. Recent publications reviews on CWs

1.2.2. Classification of constructed wetlands

CWs are classified according to the characteristics of the plants included in their system and their flow patterns. According to the macrophytes in the system there are:

- 1. Floating macrophyte-based systems (i.e. Lemna spp., Eichornia crassipes)
- 2. Rooted emergent macrophyte-based system (i.e. *Phragmites australis, Tipha* spp.)

According to water level and flow pattern:

- a) Free water surface flow (FWSCW)
- b) Subsurface flow (SSFCW)
 - Systems with horizontal subsurface flow (HFCW)
 - Systems with vertical subsurface flow (VFCW)

Additionally, different types of CWs can be combined with each other to form hybrid systems (HCWs). A summary of the types of CWs is presented in Table 1.6.

Туре						
Water level	Free water surface		Subsurface			
Plants	Free Floating Floating* leaved* Emergent		Emergent			
Flow		Horizonta	al	Horizontal	Vertical	Hybrid: Vertical + Horizontal Horizontal +Vertical
*Usually classified as macrophyte ponds						

Table 1.6. Main types of CWs for wastewater treatment (adapted from Vymazal, 2009)

The firsts CWs were of the FWS type. In the 1970s, the construction of SSFCWs was undertaken. The FWSCs dominated in North America while the SSFCWs were common in Europe and Australia (Brix, 1994; Vymazal, 2011). During the 1970s and 1980s, the SSFCWs were of horizontal flow, whereas during 1980s and 1990s the design of VFCWs began. These traditional "passive" natural systems are the most common type of CWs, but they require considerable land area, so in some cases, (e.g. strength wastewaters or to achieve higher pollutant removal performances) "intensified systems" have been developed such as aerated subsurface-flow CWs (Nivala *et al.* 2013) or baffled SSFCWs (Tee *et al.*, 2012). The main advantages and drawbacks of the CW systems are listed in Table 1.7

Advantages	Drawbacks
Good environmental integration Good elimination of the organic matter and suspended solids	The design surface area is larger than in conventional systems (especially free flow systems), although lower than in the case of ponds (especially those of subsurface flow)
Adapts well to seasonal variations in population	Risk of presence of insects (especially in those of surface flow) or rodents
No energy consumption if allowed by topography	If the elimination of suspended solids in the primary pretreatment is not efficient, clogging may appear (especially in CW of horizontal subsurface flow)
In systems of subsurface flow there may be minimal odour problems	Few control factors during operation
Easy to operate and low operating cost	Not explicit maintenance knowledge

Table 1.7. Main Advantages and drawbacks of CWs

The different types of emergent macrophyte-based systems are described in next sections.

1.2.3. Free water surface constructed wetlands

These CWs consist of basins or channels with soil or other suitable mediums to support the emerging vegetation (*Phragmites* spp, *Typha* spp., *Scirpus* spp. or *Carex* spp.) and water flowing at relatively shallow depths through the unit (Figure 1.2.).



Figure 1.2. Schematic of FWSCWs (Tilley et al., 2014)

The shallow water depth, low flow velocity, and presence of plant stalks and litter regulate water flow and, especially in long narrow channels, ensure plug flow conditions (Reed *et al.*, 1995). As wastewater passes through, it is treated through the processes of sedimentation, filtration, oxidation, reduction, adsorption, and precipitation (US EPA, 2000). Particulated organic matter tends to settle and be trapped in the system; so that they enter the biogeochemical element cycles within the water column and surface soils of the wetland. At the same time, dissolved elements enter overall mineral cycles of the wetland system. FWSCWs attract wildlife, namely insects, molluscs, fish, amphibians, reptiles, birds and mammals, as they closely resemble natural wetlands (Kadlec and Knight, 1996).This type of constructed wetland is particularly efficient in pathogen removal, due to the high exposure of the wastewater to the UV component of sunlight. Therefore, and also due to their high denitrification capacity, these systems are often used as tertiary treatment (polishing stage). The most common application is for polishing effluents from secondary treatment process (e.g. ponds, trickling filters, activated sludge, etc.).

1.2.4. Subsurface flow constructed wetlands

SSFCWs are also known as planted soil filters, reed bed treatment systems, vegetated submerged beds, vegetated gravel-bed and gravel bed hydroponic filters. A subsurface flow constructed wetland is a basin filled with some sort of filter material (substrate), usually sand or gravel, and planted with vegetation that tolerates saturated conditions. Wastewater is introduced into the basin and flows over the surface or through the substrate, and is discharged out of the basin through a structure that controls the depth of the wastewater in the wetland. The gravel or sand used in SSFCWs contributes to the treatment processes by providing a surface for microbial growth and by supporting the adsorption and filtration processes. This results in lower area demand and higher treatment performance per area for SSFCWs, as compared to FWSCWs. SSFCWs are more suitable in warmer climates because biological decomposition rates decrease with decreasing water temperature, and they potentially freeze in cold climate. In addition, the oxygen transfer from the atmosphere decreases when ice covers open water surfaces, further decreasing oxygen dependent treatment processes (US EPA, 2000).

1.2.4.1. Removal mechanisms

Pollutants removal in SSFCWs is a complex process that depends on a variety of mechanisms, including the following physical, chemical and biological processes (Reed *et al.,* 1995; Cooper *et al.,* 1996; Kadlec and Knight, 1996; Faulwetter *et al.,* 2009; Huertas, 2009; Bresciani and Masi, 2013):

- Filtration (particulated pollutants mechanically filtered as water passes through substrate and roots).
- Sedimentation (gravitational settling of solids).
- Adsorption (intermolecular force).
- Chemical precipitation (formation or co-precipitation with insoluble compounds).
- Chemical adsorption (adsorption onto substrate and plant surface).
- Chemical decomposition (decomposition or alteration of less stable compounds by phenomena such as UV irradiation, oxidation and reduction).
- Volatilization (volatilization of ammonia).
- Bacterial metabolism (removal of colloidal solids and soluble organics by suspended, benthic, and plant supported bacteria. Bacterial nitrification and denitrification).

- Plant metabolism (organic uptake and metabolism by plants. Root excretions may be toxic to enteric organisms).
- Microbiological depredation of pathogen microorganisms.
- Plant adsorption (under proper conditions, significant quantities of nitrogen, phosphorus, heavy metals or refractory organics will be taken up by plants).
- Natural die-off (natural decay of organisms in an unfavourable environment).

Table 1.8 summarises the main pollutant removal mechanisms of SSFCWs.

Pollutant	Removal mechanism				
Suspended Solids Sedimentation					
Aerobic microbial degradation Organic matter Anaerobic microbial degradation					
Nitrogen	Ammonification-nitrification-denitrification Root uptake Adsorption (adsorption in the substrate) Ammonia volatilization				
Phosphorous	Adsorption Root uptake Precipitation with cations				
Metals	Adsorption and cation exchange Complexation Precipitation Plant uptake Microbial redox				
Pathogenic microorganisms	Sedimentation Filtration Predation UV degradation Adsorption Die-off Action of antibiotics released by roots				
Organic micro-pollutants (e.g. pesticides, trichlorethane, chloroform, etc.)	Adsorption Sedimentation Volatilization Evaporation Photosynthesis Biotic/abiotic degradation				

Table 1.8. Main pollutant removal mechanisms of SSFCWs

Many reviews and studies have been conducted on pollutant removal mechanisms in SSFCWs: for organic matter (*Imfeld et al.*, 2009; Vymazal and Kröpfelova, 2009), nitrogen (Zhu *et al.*, 2010), phosphorus (Molle *et al.*, 2005; Johannesson *et al.*, 2015), metals (Marchand *et al.*, 2010), disinfection (Huertas, 2009; Sasa, 2014) and emergent pollutants (Avila, 2013).

1.2.4.2. General design and operational criteria

The design of SSFCWs is often carried out using the black box approach. Current state of the art is "semi-empirical". The systems must be individually designed for a particular set of objectives and constraints. The aim is to maximise contact between the polluted water column and various wetland components, such as biofilms, plants, the sediment layer, etc. The efficacy of contact is related, among others, to the water's flow path in the system, which in turn, is related to both the physical dimensions and retention time. Designing CWs for the treatment of pollutants initially involves the sizing of a specific wastewater flow rate, mass loading, and desired removal of a given pollutant. Currently, there are numerous manuals on the design of SSFCWs (eg. Cooper *et al.,* 1996, Kadlec and Knight, 1996; US EPA 2000; García and Corzo, 2008; UN Habitat 2008; Bresciani and Masi, 2013, Stefanakis *et al.,* 2015). In terms of general design and operational parameters, SSFCWs are defined empirically based on previous experiences (Caselles, 2007). The most important design and operational parameters that can affect removal efficiency in SSFCWs are described below.

- a) Design parameters
 - Influent quality. Influent quality depends on the type of wastewater (urban, industrial, etc.) and the absence or presence of pre-treatment. The type of pre-treatment is a key parameter for the efficiency of pollutant removal in SSFCWs and its influence should be subject to further investigation (Avila, 2013). The particular characteristics of some wastewaters (different biodegradability, toxic compounds, C/N ratios, salinity, etc.) can affect the performance of CWs and thus the removal processes and efficiency. Hence, in these cases different treatment designs and strategies are necessary (Wu et al., 2015).
 - Media characteristics. A wide range of materials and the size of the SSFCW main bed media materials have been applied globally, and its selection is often dictated by the availability, price and local practices of a certain region (Avila, 2013). The hydraulic conductivity of a porous media is quite sensitive to its size, as well as particle size distribution and particle shape (Knowles *et al.*, 2011). The choice of the medium filter size must be a technical compromise in regards to the granulometry of the gravel or sand. It must be

fine enough to ensure the retention of solids and coarse enough to prevent clogging (Boutin *et al.*, 2006).

- Media and water depth. Several studies have clearly demonstrated that water depth in the HFCWs beds constitutes a key design parameter affecting redox condition, oxygen supply and the subsequent removal efficiency of constructed wetlands (García and Corzo, 2008). Shallower depths appear to have higher redox potential and thus tend to promote greater variaty and energetically favourable reactions. Filter depth is an important factor in the removal of some pollutants of vertical attached growth in fine media, such as infiltration-percolation systems Folch (1999). Brissaud *et al.*, (1999) found that the greater the depth of the bed, the better the removal of the bacterial indicators.
- Plants. The plants used in SSFCWs are macrophytes. Commonly found plant species in the CW include the common reed (*Phragmites australis*) and cattail (Typha angustifolia), but the list is guite long (Vymazal, 2013). Of the macrophytes, *Phragmites australis* or *communis* is the most frequently used on a global level, due to its optimal performance, its ability to develop deep roots (0.5-0.7 m) and its resistance to aggressive wastewaters and disease. The role of the plants and the effect of plant type on the performance of the SSFCWs are not fully clear. Some studies have suggested that the plant species does not have much effect on SSFCW performance, particularly in VFCWs (Stefanakis and Tsihrintzis, 2012). However the role of vegetation in CWs has been found to be of great importance since the plants participate in the assimilation of nutrients, provide surface for biofilm growth, pump and provide oxygen to the to the rhizosphere and to the underground area of the systems, retain suspended particles, favour hydraulic retention time and, therefore, the processes of pollutant removal contribute directly to microbial removal through the emission of toxic substances from the roots, control algae development and protect from low temperatures (Avila, 2013; Bresciani and Masi, 2013). Although plant uptake represents a relatively small percentage of the total nutrient removal (there is a direct removal contribution in the order of 10-20% during the vegetative season), plants play a major role in enhancing nitrification and denitrification activities due to root-zone aeration and organic matter supply. Furthermore, everybody agrees to the importance of

plants in promoting the development of differentiated natural habitats and contributing to landscape value.

Part of the macrophyte	Role in treatment process
	Light attenuation > reduced growth of phytoplankton
	Influence on microclimate > insulation during winter
Aerial plant tissue	Reduced wind velocity > reduced risk of re-suspension
	Aesthetically pleasing appearance of the system
	Nutrient storage
	Filtering effect > filter out large debris
	Reduced current velocity > increase rate of sedimentation,
	reduced risk of resuspension
Plant tissue in water	Provide surface area for attached biofilms
	Excretion of photosynthetic oxygen > increased aerobic
	degradation
	Nutrient uptake
	Stabilizing the sediment surface > decreased erosion
	Prevent clogging of the medium in vertical flow systems
Roots and rhizomes in the sediment	Provide surface area for attached biofilms
	Oxygen release increases degradation (and nitrification)
	Nutrient uptake
	Release of antibiotics

Table 1.9. Role of the plants in the CWs (modified from Bresciani and Masi, 2013)

- <u>Temperature.</u> Several authors have repeatedly observed (mainly for nitrogen removal) the effect of treatment performance of constructed wetlands depending on the temperature (e.g. Stefanakis and Tsihrintzis, 2012). Moreover, temperature is one of the design parameters used when sizing HFCWs.
- b) Operational parameters
 - <u>Hydraulic load (HL).</u> HL or hydraulic loading rate (HLR) is the flow applied to the surface of the filter per unit time. It is normally expressed in m/day or cm/day. The HL is inversely proportional to the hydraulic retention time for a given SSFCW depth, and it varies from site to site and depending on wetland configuration. The HL is one of the foremost factors in performance control for SSFCWs (Toscano *et al.*, 2009; Saeed and Sun, 2012). Molle *et*

al., (2006) examined the effect of HL on VFCWs in France and Sasa (2014) also studied the effect of the HL on HFCWs.

- <u>Organic loading rate (OLR)</u>. OLR or organic load depends on the inlet quality and the HL. OLR in SSFCWs is expressed in grams of COD or BOD₅ per area (m²) per time (day). The US EPA itself, in its manual on CWs for municipal wastewater treatment (US EPA, 2000), recommends the use of area per gram of COD as a "conservative" approach to ensure reliable functioning and to respect the established concentration limits. VFCWs can accept higher OLR than HFCWs.
- Dosing and feeding regime. SSFCWs can operate continuously (usually by gravity) or intermittently (doses). The HFCWs usually operate continuously and the VFCWs are often intermittently loaded. This may influence the hydraulics of the beds as well as the oxygenation, thereby affecting the removal processes. When intermittently feeding the VFCWs, the number and quantity of doses per day may also affect the performance (Molle *et al.*, 2006). SSFCWs can also be operated with feeding/resting periods. The application of resting periods (as explained for the IP technology) can also affect the oxygenation in the bed, as well as the biomass growing. Therefore, the application of feeding/resting cycles may reduce clogging and affect performances of the SSFCWs. Sasa (2014) studied the effects of the feeding and resting periods in HFCWs. Operating HFCWs with resting periods enhances removal of some parameters.

1.2.4.3. Horizontal flow constructed wetlands

In HFCWs, wastewater is fed in at the inlet and flows slowly through the porous medium under the surface of the bed in a more or less horizontal path until reaching the outlet zone where it is collected prior to exiting via level control arrangement at the outlet (Figure 1.3.). During this passage, the wastewater will enter into contact with a network of aerobic, anoxic and anaerobic zones. The aerobic zones are found around the roots and rhizomes that leak oxygen into the substrate (Cooper *et al.*, 1996).



Figure 1.3. Schematic of HFCWs (Tilley et al., 2014)

The reactor is mainly anaerobic, with complex physical, chemical and biological mechanisms: bacterial reduction and oxidation, filtration, settling and chemical settling. Water flows underground with theoretical plug-flow, passing through the porous support media and contacting the biofilm formed over the support and plant roots. Hydraulic retention times (HRT) vary from a few to several days, depending on the management and objectives. HFCWs consist basically of:

- 1. An inlet pipe.
- 2. An outlet pipe with water level control (e.g., adjustable standpipe).
- 3. A clay or synthetic (HDPE or PVC) liner.
- 4. Filter media:
 - a. Treatment zone: the bed filling material is sized to offer an appropriate hydraulic conductivity (the most frequently used media are coarse gravel, fine gravel and coarse sand) and to furnish a large available surface for the biofilm growing.
 - b. Distribution and collection zone: the inlet and outlet zones use a large filling material, such as stones, in order to ensure easy cleaning in the case of clogging.
- 5. Emergent vegetation. The most common macrophyte is *Phragmites australis* (reeds) but *Typha* spp. (cattail) and *Scirpus* spp. (bulrush) are also used.

The sizing of the HFCWs systems depends on many parameters that should be examined during the preliminary feasibility assessment. After defining the treatment goals and the most appropriate treatment scheme, the sizing procedure may be performed using well known and scientifically approved methods. Area requirements are determined based on design equations such as the various commonly used first order kinetic equations (Reed *et al.*, 1995; Kadlec *et al.*, 1995; Cooper *et al.*, 1996) for the pollutants removal and the Darcy law for the hydraulic aspects (UN Habitat, 2008; Bresciani and Masi, 2013).

As an alternative and simpler method, it is possible to use "rule of thumb" approaches for the design, based on areal coefficients such as "area per PE" and "area per gram of COD". To reduce clogging, some authors have recommended limiting organic load rates to 6 g BOD₅/m²·day for HFCWs (García and Corzo, 2008). Until now, only simple deterministic models could be calibrated for the prevision of performances assuming the horizontal subsurface flow system as a plug-flow reactor and applying the first-order removal equation.

1.2.4.4. Vertical flow constructed wetlands

VFCWs are wastewater treatment systems with macrophytes rooted in a gravel or sand (substrate) bed that is usually of 0.6 to 1 m depth. VFCWs differ from the horizontal ones in terms of feeding method, water flow direction and filling media (Figure 1.4). Water is often applied discontinuously on the surface via several mechanisms (such as aerial pipes), infiltrates and percolates with theoretical plug-flow, passing through the porous support media and contacting the biofilm found over the support and plant roots. The new batch is fed only after all of the water percolates and the bed is free of water. This enables the diffusion of oxygen from the air into the bed. Oxygen diffusion from the air through the intermittent dosing system contributes in a larger way to filtration bed oxygenation as compared to oxygen transfer through the plant (Salgot and Torrens, 2008).



Figure 1.4. Schematic of VFCWs (Tilley et al., 2014)

The reactor is mainly aerobic, and utilizes complex physical, chemical and biological mechanisms: bacterial oxidation, filtration, physical and chemical settling, including nitrification. As a result, VFCWs are far more aerobic than HFCWs and provide suitable conditions for nitrification. On the other hand, VFCWs do not provide denitrification. HRTs are usually only a few hours, but a small part of the infiltrated water can be retained for more time. VFCWs are also very effective in removing organic particles and suspended solids. The removal of pathogen indicators is variable. The capacity of HFCWs to remove indicator microorganisms, mainly bacterial indicators, has been thoroughly examined. However, the effectiveness of disinfection and the mechanisms involved in vertical flow constructed wetlands (VFCWs) are still quite unknown.

VFCWs accept greater loads per m² than HFCWs. So, compared to HFCWs, vertical systems require less land per PE. However, the VFCWs have higher operation and maintenance requirements due to the need to pump the wastewater intermittently on the wetland surface. A VFCW typically consists of:

1. Inlet devices (feeding and distribution system). Depending on the terrain, different options are given: the height difference between the pre-treated wastewater and the bed allows for the use of mechanical devices without the need for electrical, fossil or solar energy. These energy-less systems are siphons or tipping buckets. The intermittent feeding device may be switched either by quantity, time or both. Water is then usually distributed by networks of perforated pipes.

- 2. Simple outlet pipe (drainage pipes) with no water level control.
- 3. A clay or synthetic (HDPE or PVC) liner.
- 4. Filter media: the bed filling material is sized to offer an appropriate hydraulic conductivity (the most frequently used media are fine gravel and coarse sand) and to furnish a large available surface for the biofilm growing. There is a drainage layer at the bottom with coarse gravel, and sometimes, an intermediate layer between the filtering layer and the drainage layer. The drainage may be achieved either with drainage pipes and/or with coarse gravel. In cold climates, a shallow gravel cover is recommended for the main sand layer.
- 5. Emergent vegetation: usually *Phragmites australis*.

Sizing VFCWs is usually done by calculating an area coefficient per PE (Table 1.10). They work with theoretical inlet concentrations and loads and treatment goals that are defined by the specific requirements. Sizing procedure for VFCWs beds is usually based on the nitrification process; in fact, when the normally required treatment goals for ammonium concentration are fulfilled, all of the other parameters are satisfactory eliminated as well. However, investment costs tend to be higher due to the conservative aspects of this approach.

Reference	Equation	Observations
Cooper <i>et al.</i> , (1996)	A (m ²) = 1.0PE A (m ²) = 2.0PE	Only BOD₅ removal BOD₅ and N-NH₄ ⁺ removal
Grant and Griggs (2001)	A (m ²) = 5.25PE ^{0.35} + 0.9PE	
Weedon (2003)	A (m ²) = $5.4PE^{0.6}$ A (m ²) = $2.4PE^{0.85}$	Up to 25 PE More than 25 PE

Table 1.10.	Area	coefficients	for	sizing	VFCWs
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One upper limit for the performance of VFCWs as well as HFCWs is clogging, as in the case of VFCWs on the surface. Soil clogging appears when the conductivity of the filter media is reduced. The increase of biomass and development of biofilms and microorganisms leads to a strong reduction of oxygen presence in the lower layer and a resulting decrease in efficiency yields for all of the oxidizing processes (nitrification, carbon oxidation, pathogen removal). German results have shown that good performances of VFCWs dimensioned according to the German guidelines (ATV-A

262, 1998) can be achieved over the long term using the two following empirical values: OLR= 20 g COD/m²·day with a maximum SS concentration of 100 mg/L, SS Loading Rate = 5 g/m²·day, HL <8 cm/day in winter and 12 cm/day in summer.

Usually, VFCWs are down-flow systems as in the case of IP technology. However there are some experiences with up-flow systems where water flows from the bottom of the filter to the top (Fonder and Headley, 2013). A specific VFCW configuration was developed in France and it is explained in detail below.

French vertical flow constructed wetlands were developed by Cemagref (now Irstea) over 20 years ago (Lienard et al., 1987), and were applied by the SINT company during the 1990's. French VFCWs have become the main systems implemented in small communities under 2000 PE in France (Molle, 2014). More than 2500 plants are in operation for the treatment of domestic wastewater (up to 4500 PE). Most of these plants have been built according the classical French design of two VFCWs stages, having well known guidelines and performance (Molle et al., 2005). The particularity of this system is that it accepts raw sewage directly onto the first stage allowing for easier sludge management as compared to dealing with primary sludge from an imhoff settling/digesting tank. These CWs operate like the IP systems: they are fed intermittently with loading and resting periods. The feeding with raw wastewater causes the accumulation of a layer of solids on the top of the bed, which in turn acts as a filter. The alternation of cycles of feeding and resting promotes mineralization of the solid deposits during the resting phases (Molle et al., 2006). The feeding of the filters in hydraulic batches (by a storage and high capacity feeding system) ensures an optimum distribution of wastewater across the entire infiltration area and improves oxygen renewal. The flow of raw sewage (over the short dosing period) onto the first stage must be greater than the infiltration speed (infiltration rates) in order to correctly distribute the sewage over the entire bed surface.

The deposits accumulating on the surface contribute to reduce the intrinsic permeability of the media and thus improve the distribution of wastewater. Plants limit surface clogging, since the stems pierce the accumulated deposits. When the difference in height between the inlet and outlet of the plant is sufficient, the plant operates without an energy source thanks to siphons. A scheme of the french vertical flow VFCW is shown in figure 1.5



Figure 1.5. Schematic of french VFCW (modified from Boutin et al., 1998)

The granulometry of the filters differs depending on the stage: the media for the first stage consists of several gravel layers. The primary layer is fine gravel (approximately 2-8 mm). The second stage is made up of a layer of calibrated sand having the same granulometry as in the infiltration-percolation systems. The sizing of the filters is based on an acceptable organic load, expressed as a filter surface unity per PE. (Table 1.11) Current recommendations are two stages of filters, the first of which is divided into three filters and the second into two filters.

Type of wastewater	Equation	Observations
Raw wastewater (first stage)	A (m ²) = 1.2 PE A (m ²) = 1.5 PE	Separate sewerage system Combined sewerage system
Treated wastewater	A (m ²) = 0.8 PE	Separate sewerage system
(second stage)	A (m ²) = 1.0 PE	Combined sewerage system

Table 1.11. Area coefficients for sizing French VFCWs

In the first stage of the French VFCWs, the special design and operating conditions allow for a higher organic loading rate to be applied than in the other VFCWs: applied OLR values of up to 180 g BOD₅ /m² day and 300 g COD/m²·day (Bresciani and Masi, 2013). This configuration has been found to permit a significant removal of COD, SS and almost complete nitrification (Molle *et al.*, 2005).

1.2.4.5. Hybrid systems

Hybrid systems are a combination of the two CW types. As explained previously, HFCWs and VFCWs both have different performance efficiencies, removal mechanisms, advantages and disadvantages. HFCWs have good removal efficiencies for organic matter and SS for secondary wastewater treatment, but not for nitrification due to their limited oxygen transfer capacity. Thus, there has been a growing interest in VFCWs due to their much greater oxygen transfer capacity and considerably lower surface area requirement as compared to the HFCWs. Moreover, VFCWs nitrify the influent. For these reasons, there has been a growing interest in combined (hybrid) wetlands (UN Habitat, 2008). In these systems, the advantages and disadvantages of both systems may be combined to complement each other. Depending on the needs, hybrid wetlands could be either HFCW followed by VFCW wetland or VFCW wetland followed by HFCW wetland (e.g. if nitrification-denitrification is desired).

Hybrid systems were developed in the 1960s but their use increased only in the late 1990s and the 2000s, mainly due to the stricter discharge limits for nitrogen and the more complex wastewaters treated in CWs. The early hybrid CWs consisted of several stages of vertical flow followed by several stages of horizontal flow beds. During the 1990s, HF-VF and VF-HF hybrid systems were introduced. The VF-HF hybrid constructed wetlands were mainly designed to treat domestic or municipal wastewater where nitrified effluents were required but there was also application to other types of wastewater (Vymazal, 2014).

1.2.5. Applications of constructed wetlands

CWs are commonly used to treat municipal and domestic (single house or group of houses) wastewaters as both secondary and tertiary treatment stages. The most available sets of monitoring data (such as the North American Database, the UK Constructed Wetland Association Database, several European collections, etc.) are related to this application.

Combinations or integrations of CWs targeting other special purposes are being used on a more frequent basis. With the deteriorating environment leading to stricter discharge standards, including the emphasis on effluent reuse, one type of natural technology operating as the unique treatment system may be insufficient to meet the requirements of these new more stringent guidelines, despite improvements in design and operational strategies. Therefore, there may be the need for treatment systems that integrate various types of treatment technologies in order to achieve enhanced treatment efficiency or extended treatment goals. These combined/integrated treatment systems could present a new means of tackling the individual drawbacks while improving their existing functions. The combination of CWs with other processes, such as, for example, irrigation reuse or algae control, may be developed thanks to the specific advantages of CWs, including their effective nitrogen and bacteria/pathogen removal, etc. (Xu *et al.*, 2015).

Although CWs are commonly used to treat municipal or domestic wastewater, the application of CWs has significantly expanded to the treatment of other types of wastewater including wastewater from industries, agricultural activities, runoff, etc. (Wu *et al.*, 2015).

The use of CWs to treat industrial wastewater has increased significantly over the past ten years (Rossmann *et al.*, 2013). Industrial wastewater composition differs considerably from that of municipal sewage and is quite variable in itself. Unlike municipal wastewater effluents, which usually have a similar composition, industrial wastewater tends to have a variety of components with varying degrees of biodegradability and toxicity, thus requiring different treatment designs and strategies. In many industrial wastewaters the concentrations of organics, suspended solids, ammonia or other pollutants are quite high and therefore, the use of CWS nearly always requires some sort of pre-treatment. Wu *et al.* (2015) presents full-scale cases of CWs treating various industrial effluents and the challenges and strategies related to treatment of these types of wastewater. The challenges include high organic loading, salinity, extreme pH, low biodegradability and colour. Vymazal (2014) summarizes 138 CWs installations in 33 countries treating a total of 26 types of industrial wastewaters.

Another application of CWs is the treatment of agricultural wastewater. Wastewater from intensive agro-industrial activities (e.g. pig farms) contains significantly higher concentrations of organic matter and nutrients than municipal effluent. These high pollution loads may contribute to water management problems if waste is allowed to discharge directly into receiving water. Wastewater from these agro-industries is often accumulated in ponds, which act as the initial phase of treatment. There are some cases of CWs treating this type of wastewater (Vymazal and Kröpfelová, 2008). There is even a piggery wastewater treatment wetland database available (Politeo, 2013), including 13 CWs sites.

A recently developed application of constructed wetland is related to diffuse (runoff) pollution treatment. Several kinds of diffuse pollution, such as agricultural, urban or infrastructures runoff may be addressed using CWs, in which the effective removal of nutrients and micro-pollutants, such as persistent organic compounds (i.e. polycyclic aromatic hydrocarbons generated by vehicles' fuel engines) makes these techniques quite suitable for watershed scale approaches in cases in which specific local treatment in inappropriate. Finally, other specific applications include the treatment of landfill leachates. A summary of the CWs applications is shown in Table 1.12.

Secondary treatment of domestic wastewaterUrban wastewaterSecondary treatment of municipal wastewaterTertiary treatment as polishing stage in conventional treatment plants or other natural systemsFood processing (slaughterhouse and meat processing, milk and cheese industry, olive mill effluents, sugar industry, potato processing, seafood processing)Industrial wastewaterFood processing (slaughterhouse and meat processing, milk and cheese industry, olive mill effluents, sugar industry, potato processing, seafood processing)Industrial wastewaterPetrochemical Tannery TextileIndustrial wastewaterTannery TextileAgroindustrial wastewaterPig farms Dairy farms Fish farmsAgroindustrial wastewaterAirports Agricultural Urban and highway Greenhouses Plant nurseries	Type of wastewater/waste	Applications			
Urban wastewaterSecondary treatment of municipal wastewaterTertiary treatment as polishing stage in conventional treatment plants or other natural systemsFood processing (slaughterhouse and meat processing, milk and cheese industry, olive mill effluents, sugar industry, potato processing) Petrochemical Pulp and paper industry Tannery TextileIndustrial wastewaterTannery Textile Fish and shrimp aquaculture Alcohol fermentation industry (winery, distillery) Laundry Chemical industryAgroindustrial wastewaterPig farms Fish farms Fish farmsRunoffUrban and highway Greenhouses Plant nurseriesRunoffLandfill leachate		Secondary treatment of domestic wastewater			
Tertiary treatment as polishing stage in conventional treatment plants or other natural systems Food processing (slaughterhouse and meat processing, milk and cheese industry, olive mill effluents, sugar industry, potato processing, seafood processing) Petrochemical Pulp and paper industry Tannery Textile Fish and shrimp aquaculture Alcohol fermentation industry (winery, distillery) Laundry Chemical industry Agroindustrial wastewater Pig farms Fish farms Fish farms Airports Agricultural Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate	Urban wastewater	Secondary treatment of municipal wastewater			
cr other natural systems Food processing (slaughterhouse and meat processing, milk and cheese industry, olive mill effluents, sugar industry, potato processing) Seafood processing) Petrochemical Pulp and paper industry Petrochemical Pulp and paper industry Tannery Textile Fish and shrimp aquaculture Alcohol fermentation industry (winery, distillery) Laundry Chemical industry Pig farms Fish farms Fish farms Agroindustrial wastewater Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate		Tertiary treatment as polishing stage in conventional treatment plants			
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cheese industry, olive mill effluents, sugar industry, potato processing, seafood processing) Petrochemical Pulp and paper industry Tannery Tannery Textile Fish and shrimp aquaculture Alcohol fermentation industry (winery, distillery) Laundry Chemical industry Pig farms Dairy farms Fish farms Fish farms Fish farms Agricultural Runoff Urban and highway Greenhouses Plant nurseries		Food processing (slaughterhouse and meat processing, milk and			
seafood processing) Petrochemical Pulp and paper industry Tannery Tannery Textile Fish and shrimp aquaculture Alcohol fermentation industry (winery, distillery) Laundry Chemical industry Pig farms Dairy farms Fish farms Fish farms Agroindustrial wastewater Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate		cheese industry, olive mill effluents, sugar industry, potato processing,			
Petrochemical Pulp and paper industry Tannery Tannery Textile Fish and shrimp aquaculture Alcohol fermentation industry (winery, distillery) Laundry Chemical industry Obiry farms Fish farms Fish farms Agricultural Runoff Urban and highway Greenhouses Plant nurseries		seafood processing)			
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Textile Fish and shrimp aquaculture Alcohol fermentation industry (winery, distillery) Laundry Chemical industry Pig farms Dairy farms Fish farms Agrouttural Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate	Industrial wastewater	Tannery			
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Alcohol fermentation industry (winery, distillery) Laundry Chemical industry Pig farms Dairy farms Fish farms Airports Agricultural Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate		Fish and shrimp aquaculture			
Laundry Chemical industry Pig farms Dairy farms Fish farms Airports Agricultural Runoff Urban and highway Greenhouses Plant nurseries		Alcohol fermentation industry (winery, distillery)			
Chemical industry Pig farms Dairy farms Fish farms Airports Agricultural Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate		Laundry			
Agroindustrial wastewaterPig farmsAgroindustrial wastewaterDairy farmsFish farmsFish farmsAirportsAgriculturalRunoffUrban and highwayGreenhousesPlant nurseriesLandfill leachateLandfill leachate		Chemical industry			
Agroindustrial wastewater Dairy farms Fish farms Airports Agricultural Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate		Pig farms			
Fish farms Airports Agricultural Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate	Agroindustrial wastewater	Dairy farms			
Airports Agricultural Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate		Fish farms			
Agricultural Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate		Airports			
Runoff Urban and highway Greenhouses Plant nurseries Landfill leachate		Agricultural			
Greenhouses Plant nurseries Landfill leachate	Runoff	Urban and highway			
Plant nurseries Landfill leachate		Greenhouses			
Landfill leachate		Plant nurseries			
		Landfill leachate			
Others Abandoned and active mine drainage	Others	Abandoned and active mine drainage			
Sludge dewatering and mineralization		Sludge dewatering and mineralization			

Table 1.12. CWs applications

2. OBJECTIVES

2. OBJECTIVES

The **general objective** of the thesis is to examine the viability of SSFCWs for the treatment of wastewater derived from different sources (<u>wastewater treatment</u> <u>ponds/lagoons</u>, <u>pig farms</u> and <u>car wash facilities</u>) for different purposes and to evaluate the influence of design and operational parameters on treatment efficiency and hydraulic behavior.

- (a) <u>Wastewater treatment pond effluents</u>. The aim of this study is to evaluate the viability of VFCWs and HFCWs to treat the effluent from wastewater treatment pond systems for reuse or discharge in water bodies.
- (b) <u>Pig farm effluents</u>. The aim of this study is to evaluate the viability of hybrid SSFCW to treat swine slurry for and application or discharge in water bodies.
- (c) <u>Car wash effluents.</u> The purpose of this study is to evaluate the viability of VFCWs and HFCWs to treat the effluent from car wash facilities for their recycling, in order to reduce tap water consumption.

The specifics objectives are:

- To characterise the different wastewaters.
- To evaluate performance efficiency (in terms of physicochemical and microbiological indicators) and hydraulic behavior of the SSFCW pilots.
- To study the algae dynamics in pond effluents and their removal in SSFCWs.
- To study the capacity of nitrification/denitrification of a hybrid system treating partially settled swine slurry.
- To study the occurrence of specific pollutants in the car wash effluents and their removal in SSFCWs compared to an IP system.
- To examine the influence of design parameters (size and type of media, presence of *Phragmites australis*, media depth) and operational parameters (hydraulic load, dosing and feeding modes) on the treatment efficiency and hydraulics of the SSFCWs.
- To study the role and influence of deposits on the surface of the VFCWs.
- To determine the drawbacks of SSFCWs for each type of wastewater.

The experimental studies of this thesis were conducted within the framework of different R+D+I projects.

The first study site to upgrade the effluent quality of wastewater treatment ponds was coordinated by CEMAGREF-Lyon (now IRSTEA) and took place in the Aurignac WWTP (France), under the framework of the European project LILIPUB (LIFE3-ENV-F303). Complementary studies were carried out in the WWTP of Santa Eugènia (Mallorca) as part of the European project MEDIWAT (1G-MED09-262) coordinated by Water Observatory, (Sicilian Region Service) in collaboration with AQUALOGY S.A.

The treatment of swine slurry was conducted in the small farm "Can Coromines", located in Viver i Serrateix (Barcelona), under the framework of the Spanish Ministry of Innovation and Science Program (CTM2010-19197) and the European project MEDIWAT (1G-MED09-262), in collaboration with MOIX Serveis i Obres S.L.

Finally, the studies to treat wastewater from car wash facilities took place at the car wash station of Montfullà (Girona), under the framework of the European project MinAqua (LIFE11-ENV- 569), coordinated by Fundació Ramón Noguera.

3. SUBSURFACE FLOW CONSTRUCTED WETLANDS FOR POND EFFLUENT QUALITY IMPROVEMENT

3. SUBSURFACE FLOW CONSTRUCTED WETLANDS FOR POND EFFLUENT QUALITY IMPROVEMENT

3.2. Introduction

3.1.1. Problem statement

Pond systems have been used as a preferred treatment system for many applications in developing and developed countries and are particularly suitable wastewater treatment systems for small communities. The most significant advantages of pond systems are their simplicity, low construction, operation and maintenance costs and ability to withstand hydraulic shock loadings.

Despite these advantages, variability in the quality and high concentrations of microalgae in the effluent can limit the practical applications of these systems. Algae in the effluent manifest in the form of SS and exert an oxygen demand from the receiving stream through its bacterial degradation. This has been recognized as one of the most troublesome operational problems in wastewater treatment using ponds (WEF, 1992). Pond effluent quality is sometimes inadequate in meeting the environmental objectives of the receiving waters. For example, parameters that may need improvement include pathogens, nutrients, SS and BOD₅ contents.

Some authors believe that algal parameters (SS and BOD₅) should not be subject to conventional effluent requirements (Mara, 1998). In some occasions, standards for BOD₅ and SS concentrations have been relaxed for pond system effluents (*e.g.*, in the United States, France, Germany, and the European Union). This is because algal cells are not rapidly biodegradable and have low settling velocities; thus, they do not readily settle in streams and may be dispersed over a wide area before they exert an oxygen demand on the watercourse. Furthermore, continuing photosynthesis may result in net oxygen input into a receiving water course, and the algae may promote an increase in the productivity of the aquatic system. However, uncontrolled discharge of algae at high concentrations is undesirable as it can deplete oxygen reserves (Shilton, 2006).

The European Union's relaxation of standards (for communities of more than 2000 PE) is defined in the EC Urban Wastewater Treatemnt Directive as follows: BOD₅ and COD analyses on the effluent may be carried out on filtered samples and SS must not

exceed 150 mg/L (European Union, 1991). For small communities, the European Directive 91/271/CEE established that wastewater from urban settlements with less than 2000 PE must be treated in an "adequate way", thus protecting the receiving body. Although this Directive does not specify specific quality parameters for pond effluents, regulators of most of European countries have set stricter standards than those defined by the European Union, depending on the receiving body. Therefore, pond effluent quality is sometimes inadequate to meet the environmental objectives of policies regarding the receiving waters (parameters that may need improvement include concentrations of SS, COD, BOD₅, bacterial indicators, nutrients and ammonia) (*Torrens et al.*, 2006a). Studies conducted in France showed that in the case of sensitive receiving bodies, the pond effluent quality was often insufficient according to the predetermined limits (Racault and Boutin, 2005).

Additionally, these algae can also impose serious constraints for some potential areas of effluent reuse, *e.g.*, agricultural applications (Saidam et al., 1995). Large quantities of algae in pond effluents intended for use in agricultural irrigation can cause problems for irrigation infrastructure networks, especially in low-flow drip-irrigation systems, where physical blockages can occur. Moreover, as environmental pollution and reuse standards have become more stringent, traditional pond systems have become increasingly inadequate in many instances, particularly with regards to effluent quality. Because of the stringent legislations in terms of reclaimed water reuse, ponds effluent cannot be always be reused (Kaya *et al.*, 2007).

These drawbacks have caused many communities to either choice to other treatment systems or to link pond systems to other complementary wastewater treatment systems to upgrade the pond effluent. Several technologies have been proposed for upgrading pond effluent quality, which will be described in section 3.1.3.

3.1.2. Characteristics of wastewater pond effluents

The performance of wastewater treatment ponds depends on the effective use of bacteria for the degradation of organic matter, efficient use of algae for maintaining an adequate level of oxygen in the system and separation of algal biomass from the effluent (WEF, 1992). Excessive loss of algae in a pond deteriorates the quality of the effluent. When proper hydraulic residence times are not provided for ponds, the organic matter contents in the effluents can be higher than those of the influents. By their nature, facultative ponds produce large quantities of algae. Under normal operating

conditions, over 90% of the suspended solids that leave a facultative pond during the summer are due to algae.

As an index of the effect that algae in suspension of a pond effluent has on some parameters, rough approximations have been suggested. Pearson and König (1986) found that a linear relationship exists between the concentrations of algal chlorophyll-a (Chl-a) and COD: approximately 1 mg of algal Chl-a is equivalent to 300 mg of COD in pond effluent, although these values can vary with algal genera. Mara *et al.* (1992) reported that the algal concentrations in facultative pond effluents ranged between 1000-1500 µg Chl-a/L in hot climates and could reach these concentrations in temperate zones during the summer, where algal contribution to SS concentrations could reach 40-100 mg/L. According to Meiring and Oellerman (1995), 100 µg/L of Chl-a give rise to 5.6 mg of COD/L. Similarly, Shipin *et al.* (2007) reported this figure to be 10 mg/L of COD, 3 mg/L of BOD₅ and 20 mg/L of SS. The concentration of algae in a healthy facultative pond depends on the loading and temperature but is usually in the range of 500–2000 µg Chl-a/L, which represents effluent COD and SS concentrations of 28–200 mg/L and 100–400 mg/L, respectively (Kaya *et al.*, 2007).

The movement of algae can lead to diurnal fluctuations in effluent quality depending on the take-off height of the outlet. Column samples taken at effluent points can provide a good estimation effluent quality. To reduce the algal concentration leaving facultative ponds, it has been suggested that the outlet should be positioned at a depth of 40-50 cm in order to avoid part of the algal band (Shilton, 2006).

Tables 3.1 and 3.2 present the main characteristics of the influent and the effluent from pond systems in Catalonia.

		Stabilization ponds (n=7)		
Paramet	Parameter		Actual	
	Influent	260	260	
55 (mg/L)	Effluent	24	100	
	Influent	280	280	
BOD5 (Mg/L)	Effluent	26	67	

Table 3.1. Characteristics of influent and effluent water from pond systems in Catalonia (from
García *et al.*, 2000)

(n=1) 82 33	n=2)
82 33	ND
33	ND
	ND
18	ND
9.1	ND
1.4.10 ⁷	⁷ 1.4.10 ⁵
5.7.10 ⁴	⁴ 1.0.10 ⁴
2.0.10 ⁶	⁶ 2.5.10 ⁴
	³ 1.7.10 ³
6	⁶ 2.0.10 ³ 9.4.10

Table 3.2. Nutrient and fecal pollution indicators in the influent and effluent from ponds in
Catalonia (from García *et al.*, 2000)

Table 3.3 presents a summary of the European regulations concerning discharge from urban wastewater treatment plants (>2000 PE) and the average effluent quality of pond systems in France and Catalonia.

	European regulations for	Pond system			
	2005 - Minimum percentage	performances			
Parameters		France	Catalonia		
	> 2000 PE	(Racault and Boutin,	(García <i>et al</i> .,		
		2005)	2000)		
COD		162 mg/L			
dCOD	125 mg/L or 75 %	99 mg/L			
BOD ₅	25 mg/L or 75 %	43 mg/L	67 mg/L		
SS	150 mg/L or 90 %	60 mg/L	100 mg/L		
TN		22 mg/L			

Table 3.3. Summary of the European regulations concerning discharge from urban wastewater treatment plants and average performances of pond systems in France and Catalonia

The data presented in Table 3.1, 3.2 and 3.3 show that the average pond effluent quality of the studies conducted in France and Catalonia did not comply with predetermined limits for PE> 2000. In the case of sensitive receiving bodies, traditional pond configurations in urban settlements with less than 2000 PE would not meet the stringent limits (e.g., according to the limits set by regulations in France: <125 mg/L of COD and < 25 mg/L of BOD₅). Additionally, the pond effluent qualities were too low to meet the limits necessary for the majority of wastewater reuse applications.

3.1.3. Techniques for upgrading wastewater pond effluent quality

There are a number of technologies that have been proposed for use in upgrading pond effluent quality. Technologies such as centrifugation, micro-straining, coagulation-flocculation, dissolved air flotation, sand filters and rock filters have been discussed extensively in the literature (Middlebrooks, 1995, Saidam *et al.*, 1995; Alcalde, 2001; Neder *et al.*, 2002; Torrens, 2004; Johnson and Mara, 2005; Shilton, 2006; Hamdan and Mara, 2009). Trickling filters have also been studied (Kaya *et al.*, 2007).

On the other hand, harvesting of algae for use as a fertilizer, protein rich feed, biofuel or other purposes a practice that is increasing (not usually with traditional ponds but with HRAPs). HRAPs are specific types of artificial ponds where oxygen is supplied mechanically by a surface aerator or air blower and have high algae concentrations. While the use of HRAPs for biofuel production alone is not yet economically favourable, the coupling of wastewater treatment systems with biofuel production mechanisms is considered to be financially viable. Harvesting and thickening of microalgae can be achieved by means of several techniques including coagulation-flocculation, sedimentation, flotation, centrifugation, magnetic separation and electrophoresis (Park and Craggs, 2010).

Every method of algae removal from ponds has specific advantages and disadvantages, but the selected method must be specific to the particular treatment situation. It would be desirable if these post-treatment methods could also ensure that the global treatment systems maintain the primordial advantages of the natural ponds (easy operation and exploitation, environmental integration) (Torrens, 2004). Regarding natural systems, there are some previous reports on the use of macrophyte ponds, IPs and CWs for upgrading pond effluent quality (Neder *et al.*, 2002; Torrens, 2004).

IPs can upgrade pond effluent by filtering and mineralising SS. The algae remain on the surface of the sand filter as the wastewater is treated. In 1998, a survey was conducted on the design, performance and operation of waste stabilization ponds associated with infiltration percolation in France (Marechal, 1998). There were seventy-four WWTPs with this configuration. The data selected were obtained from samples from fifty-eight facilities. Some partial conclusions from the data survey interpretation were that the average total surface of the pond systems was 8.08 m²/PE (1.6-14 m²), the average total surface area of the filters was 1.4 m²/PE and 68 % of the filters had depths between 0.7 and 1.2 m. From an effluent quality point of view, the author

proved good performances in terms of COD (90 %), BOD₅ (95 %), SS (95-98 %) and TKN (90%) concentrations. However, the performances were related to under loaded filters; hence, these performances were not representative of the overall performances of IPs. A link was found between poor IP performances (particularly TKN) and bed problems (especially with regards to clogging and bad influent repartition). From this survey, Marechal concluded that there is generally not a common criteria that can be used in the design of this combination of treatments and noted the following problems when IPs were used to upgrade pond effluent: clogging, granulometry-related issues (excessive large and small grain sizes and no granulometry indications), weeds growing on the surface of the filters, bad flow repartitions, irregular hydraulic regimes, excessive hydraulic loads and lack of maintenance.

Macrophyte ponds are also used to upgrade pond effluents (Youngchul and Wang-Joong, 2000; Neder *et al.*, 2002; Bojcevska and Tonderski 2007). However, these studies did not conclude the viability of using floating plants to reduce algae related parameters (mainly SS).

3.1.4. Constructed wetlands experiences in upgrading pond effluent quality

CWs are one of the technologies that have been studied to upgrade pond wastewater effluent, mostly regarding subsurface flow horizontal CWs. A summary of these experiences is presented in Table 3.4

SSFCW Type	Influent HL (cm/d)	Media depth (m)	Media Granulometry (mm)	Macrophyte	Feeding regime	COD (% or mg/L effluent)	SS (% or mg/L effluent)	Other parameters	Reference
VFCW	1.8-4	0.7	Sand: 0-2 mm Gravel: 20 mm	Phragmites	Intermittent	76 %		Nitrification (%) T> 10°C \rightarrow 90% 5°C< T<10°C \rightarrow 70 % T< 5°C \rightarrow 50%	Kayser <i>et al.</i> , (2002)
VFCW	3	0.9	Two sand media Sand 1: 0.21 -0.45 Sand 2: 0.23-1.38	Typha	3 times per day	Sand 1: 25-45 % Sand 2: 12-43 %		TKN 7-67 % (Sand 1) 2- 68 % (Sand 2)	Sezerino <i>et al.</i> , (2003)
FCW	Phase 1: 2.7 Phase 2: 23	0.6-1	Gravel 1-25 mm	Typha	Continous	Phase 1: 54-74 % Phase 2: 42-59 %	Phase 1: 67-87 % Phase 2: 45-56 %	Total Coliforms Phase 1: 90 % Phase 2: 28 % Fecal Coliforms Phase 1: 91 % Phase 2: 34 %	Mashauri <i>et al.</i> , (2000)
HFCW	1.4-2	0.35	Gravel 5-8 mm	Phragmites, Scirpus, Typha	Continous	Spring/summer: 23 % Fall/winter: 45 %	Spring/summer 20 %	Chl-a Spring/summer: 18 % Fall/winter: 25 %	Gschlößl <i>et al.</i> , (1998)

 Table 3.4. SSFCWs for upgrading pond effluent quality
SSFCW Type	Influent HL (cm/d)	Media depth (m)	Media Granulometry (mm)	Macrophyte	Feeding Regime	COD (% or mg/L effluent)	SS (% or mg/L effluent)	Other parameters	Reference
HFCW	20	0.4	Natural soil	Phragmites, Scirpus	Continous	Spring/summer: 62 %	Spring/summer: 20 %	Chl-a Spring/summer: 54 % Fall/winter: 10 %	Gschlößl <i>et al</i> ., (1999)
HFCW	10	0.75	Gravel 6-55 mm	Phragmites, Typha	Continous	89.3 %		BOD ₅ 71.6 % TN 48.1 %	Senzia <i>et al.</i> , (2003)
HFCW	0.4 m ² PE (dam)	0.4	Gravel 8-16 mm	Phragmites, Phalaris, Typha	Continous	37 %		TN 31 % TP 16 % Chl-a 38 %	Steinmann <i>et al.,</i> (2003)
HFCW	2.8	0.5	Gravel 30 mm	Typha, Glyceria, Iris	Continous	74% (unfiltered COD)	74 %	<8 mgN/L	Jonhson <i>et al.,</i> <i>(</i> 2007)

Table 3.4 (continued) SSFCWs and their effects on upgrading pond effluent quality

Table 3.4. shows that most of the SSFCWs experiences entailed the use of HFCWs. The removal of SS, COD and Chl-a was extremely variable depending on parameters such as the type of CW, HLs, type of material, and type of plant. Moreover, the results were variable within the same experiment depending on the season (especially for the removal of chlorophyll).

3.2. Specific objectives

The purpose of this study is to evaluate the viability of using SSFCWs to treat the effluent from wastewater treatment pond systems for different ways to deal with it, either disposal or reuse. Two study sites are considered.

- The <u>specifics objectives</u> of the main study site (study site 1) are:
 - To fully characterise the facultative pond effluent.
 - To operate in parallel for 24 months six different VFCWs pilot plants that treat real pond effluent.
 - To study the algae dynamics in pond effluents and their removal in SSFCWs.
 - To evaluate and compare the treatment efficiency and hydraulic behaviour of the six filters, monitoring physico-chemical parameters, bacterial and viral indicators, algal solids and associated parameters.
 - To study the influence of design factors/parameters (presence of *Phragmites*, media depth, type of media), operational parameters (hydraulic load, dosing modes, feeding/resting periods) and deposits on the surfaces of the filters on treatment efficiency and hydraulic behaviour of the filters.
- The <u>specific objectives</u> of the complementary study site (study site 2) are:
 - To characterise the tertiary facultative pond effluent.
 - To specifically design and operate four pilot plants (two VFCWs and two HFCWs) in parallel for 15 months.
 - To evaluate and compare the treatment efficiency of the four filters, monitoring physico-chemical parameters, bacterial indicators, algal solids and associated parameters.
 - To study the influence of the design parameters (media depth for the two VFCWs, and media size for the two HFCWs) and one operational parameter (HL) on the treatment efficiency of the filters.

3.3. Material and Methods

3.3.1. Study site 1

3.3.1.1. Study site description

The Aurignac experimental WWTP is located in the Haute-Garonne Department of France. The climatic condition in Aurignac is that of temperate climate with a mean annual rainfall about 700 mm. Autumn and spring are the seasons with higher pluviometry. The mean daily air temperature varies between 1° C and 26° C. The facility was built in 2003 and was designed to serve 300 PE. A combined sewerage system is used; it collects both sewage and rainwater, which enters the wastewater treatment facility. The experimental WWTP was designed purposely for the European project LILIPUB (LIFE3-ENV-F303) coordinated by the IRSTEA–Lyon.

Wastewater either flows into the plant under gravity, or is pumped in from regulation tanks. As the wastewater enters reaches a treatment train consisting of a settling tank (ST) one facultative pond (7 m²/PE) followed by six independent filters in parallel (four unplanted VFCWs and two planted VFCWs) with a surface area of 50 m² each (1 m²/PE). From the filters, the treated wastewater is disposed of in a small stream. Part of the primary sludge is extracted from the bottom of the ST situated upstream from the pond and pumped towards four sludge dewatering reed beds (SDRBs). After percolating through the SDRBs, the treated lixiviate is returned to the ST. Figure 3.1 shows the general layout of the plant.



Figure 3.1. Layout of the Aurignac WWTP (modified from Boutin et al., 2002)

The pond outlet is a pipe located at a height of 80 cm opposite the inlet. There is a bypass at a height of 130 cm from the pond bottom; consequently the water height can fluctuate between 80 and 130 cm. This supplementary freeboard of 50 cm allows the storage of part of the storm waters drained by the combined sewerage network. This provides an additional volume of 1050 m³. In normal functioning, the effluent is sent by gravity towards the filters as soon as the level of the pond passes 80 cm. When the water level in the pond reaches a height of 1.3 m, part of the water in the pond is evacuated thanks to the by-pass towards the neighbouring stream. The physical characteristics of the pond are given in Table 3.5. The theoretical hydraulic retention time¹(tHRT) can vary from 37 to 60 days depending on the water level in the pond.

Level	Storage volume	tHRT
(cm)	(m³)	(days)
50	1680	≈ 37
80	2100	≈ 46
130	2730	≈ 60

Table 3.5. Physical characteristics of the pond and tHRT (study site 1)

¹ tHRT (theoretical hydraulic retention time)= $\frac{V}{Q_i} = \frac{A \cdot H}{Q_i}$ where Qi (design inlet flow)=45 m³/day, V=volume of the pond, A=Area of the pond, H=height of the water

3.3.1.2. Pilot plant description

After the pond there are six filters with a surface of $1 \text{ m}^2/\text{PE}$ and a total surface of 300 m². Each filter is 50 m² in area: 2 m in width and 25 m in length. The filters contain three different media (river sand, river sand with *Phragmites australis* and crushed sand). Two filter depths were used for each medium, 25 and 65 cm, in order to determine the optimum depth of the filtering beds in this treatment. To keep costs within reasonable (and allow for gravity feeding) the thickness of the sand layer was as small as possible. Under the medium in all beds there was a 5 cm transition layer of 6-15 mm gravel, and at the bottom of each bed there was a 40 cm drain layer of 30-60 mm gravel.

The river sand was chosen according to the recommendations given in France latest recommendations in France (Liénard *et al.*, 2001). River sand generally comes from river beds or alluvial areas and is called alluvial sand. Crushed sand normally comes from quarries, and is formed by crushing rocks and stones. The two sands used in this study could be compared structurally as they both had the same mineralogical characteristics because they had the same origin: the Garonne flood plain. The shape of the grains is a difficult parameter to quantify and only a qualitative appreciation can be carried out (Figures 3.1 and 3.2). In these pictures it can be appreciated the angularity of the crushed sand compared to the rounded forms of the river sand.



Figure 3.1. Crushed sand

Figure 3.2. River sand

Sand granulometry studies (particle size distribution curves) were carried out by Huynh (Huynh, 2004) (see Apendix C). A summary of the characteristics of the sands used is presented in Table 3.6. The main difference between the two sands is their uniformity coefficient, which is about twice as much for the crushed sand as for the river sand.

Characteristic		River Sand	Crushed Sand
d ₁₀	mm	0.25	0.19
CU		4.7	9.3
Fine content	%	2.1	4

Table 3.6. Sand characteristics (study site 1)

The effluent was applied via two distribution pipes located at the top of each filters. Holes, 8 mm of diameter spaced every 300 mm along the top of each distribution pipe, disperse the wastewater over the surface of the filter. The following table summarises the characteristics of each filter and indicates the names employed in the study to define each bed configuration. Figure 3.3 presents a view of the filters.

Filter			Sand Characteristics				
	Depth (cm)	Support media		CU	Fine content		
			d ₁₀	00	(% by weight)		
M25	25	Planted river sand	0.25	47	2.1		
M65	65	Planted river sand	0.25	4.7	2.1		
R25	25	River sand	0.05	4 7	0.4		
R65	65	River sand	0.25	4.7	2.1		
C25	25	Crushed sand	0.40	0.0	4.0		
C65	65 Crushed sand		0.19	9.3	4.0		

 Table 3.7. Filter nomenclature and main characteristics (study site 1)



Figure 3.3. Planted and unplanted VFCWs

3.3.1.3. Experimental protocol

The pilot plant was monitored and operated during 24 months (2004-2006). The operation conditions and monitoring are described in next sections.

3.3.1.3.1. Operation

The filters were intermittently fed by pumping the water (55m³/h) through a distribution network under pressure. The volume of each batch was managed by a water height sensor located in a feed tank. The filters were fed alternately. The two filters containing the same medium (but witth different heights) were fed at the same time for 3 or 4 days and then rested for 1 week. The operation of the filters varied over the period of study. Different HLs and dosing frequencies were tested for each filter in order to establish the impact of the operational mode on their hydraulics and performance. The daily number of dosing-drainage cycles (f) ranged from 3 to 32, depending on the HL applied and the dose size. The filters were alternately fed for 3-4 days followed by a rest period of 7 days. A summary of the operation periods is displayed in Table 3.8.

Period	Duration	н	Dosing modes				
	(months)	(cm/dav)	Height of batch	Number of batches per day			
	(monulo)	(0.11 ddy)	(cm)	(f)			
1	4	20	5	4			
2	8	40	5	8			
3	5	75-80	5	15-16			
4	5	75-80	2.5	30-32			

Table 3.8. Operation pe	eriods (study site 1)
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3.3.1.3.2. Monitoring

Water quality monitoring

<u>Physicochemical analyses.</u> Physicochemical parameters were evaluated in each component of the plant: (a) pond inlet, (b) pond outlet and (c) filter's outlet. A monitoring program consisting of analysing 24h composite samples (40 tests) and grab samples (collected every 1 or 2 weks) was performed for 24 months. Temperature, EC, pH, COD, dCOD, BOD₅, SS, TKN, N-NH₄⁺, N-NO₃⁻, P-PO₄³⁻ and TP were analyzed according to standard French

methods (AFNOR, 2005). The particle-size distribution of the suspended solids in the water samples (pond outlet) was determined using a laser particle analyser (laser granulometer Hiac Royco Pacific Scientific 8000). The analytical method's references are listed in Appendix A. The analysis were performed in the Irstea (Lyon) laboratories and in the Soil Sience laboratory of the UB.

- <u>Microbiological analyses.</u> Microbiological parameters were evaluated in each component of the plant: (a) pond inlet, (b) pond outlet and (c) filters outlet. The analysis of 24-h composite samples (30 campaigns) and grab samples (collected every 3 weeks) was performed for 24 months. The analysis of the microbiological indicators (FC, *E. coli*, somatic coliphages and F-specific bacteriophages) was performed within 24 h, following ISO (1995, 2000a, 2000b) and Standard Methods (APHA-AWWA-WPCF, 2005). The analysis method's references are listed in annex Appendix A. The analysis were performed in the Soil Sience laboratory of the UB.
- <u>Algae monitoring.</u> The quantitative and qualitative algal biomass evolution was studied in the pond filters outlet by optical microscopy and Chl-a analysis (AFNOR, 2005). The methodology for algal identification is shown in Appendix A. The analysis were performed in the Irstea (Lyon) laboratories and Soil Sience laboratory of the UB.

Deposit layer monitoring

Samples of the deposit eventually accumulated on the surface of the filters were collected in three sampling campaings to characterise the nature of the material. DM (%), and VS (%) were analyzed following the procedures described in Appendix A. Algal identification was also performed in the deposits. The analyses were performed in the Soil Sience laboratory of the UB.

Hydraulic monitoring

Inlet and outlet flows were measured using pump functioning time and Venturi channels. Infiltration Rates (IR) were quantified by measuring the level of the surface water with two ultrasound probes per filter. The water level increases during the feeding period and then decreases. During this decreasing period, the IR was

calculated in relation to the measurement period by determining the slope rate of change in the height of the water curve of the filter over time. All data was recorded each minute with a data acquisition station (ICP-BGP). The data could be accessed locally or remotely.

Periodic tracer tests (Table 3.9) were carried out on the filters using NaCl, the content of which was monitored by electrical conductivity sensors. In each test, the tracer was added to the water of only one feeding sequence. The tracer content and the flow rate at the outlet of the filters were recorded at a one minute time step. This monitoring allowed determination of the detention time distribution (DTD), DTD=E(t), and the mean hydraulic retention time (mHRT). In addition, a minimum HRT (minHRT) was calculated as the time required reaching 10% of initial salt concentration in the filter effluent. This parameter was reported by Stevik *et al.* (1999) to be more relevant for predicting microbiological removal than mHRT. The methodology used for the performance of the tracer tests as well as the calculation of the tracer tests parameters are shown in Appendix B.

Tracor tost	Filtors	HL	Height of the	Number of batches per
Tracer test	Fillers	(cm/day)	batch	day
1	R65, R25	35	5 cm	7
2	C65, C25	35	5 cm	7
3	R65, R25	40	5 cm	8
4	M65, M25	75	5 cm	15
5	R65, R25	75	5 cm	15
6	C65, C25	40	5 cm	8
7	M65, M25	40	5 cm	8
8	R65, R25	40	5 cm	8
9	R65, R25	75	2.5 cm	30
10	C25	75	2.5 cm	30
11	R25	75	2.5 cm	30

Table 3.9	. Summary	of the	tracer	experiments	(study site	1)
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<u>Temperature inside the filters monitoring</u>

Temperature inside the filters was also monitored by 6 sensors (1 per filter) located at a depth of 15 cm. Temperatures were recorded each minute with data acquisition station (ICP-BGP). The data could be locally or remotely accessed.

Weather data monitoring

Air temperature, humidity, precipitation, pluviometry, and solar irradiation was recorded each minute on a central electronic central station of data acquisition station (ICP-BGP). The data could be locally or remotely accessed.

3.3.1.3.3. Statistical analysis

Statistical analysis was performed on the raw data using the statistics computer packages Excel 2007 and SPSS 13.0 for Windows. Excel 2007 was used to carry out descriptive statistics (i.e. averages, SD) and to perform regression analysis. SPSS 14.0 was used to analyse variance (ANOVA). One level of significance (p) was established ($p \le 0.05$). Data for these test parameters that were not normally distributed were log-transformed to present the normal distribution required to run these analyses.

3.3.2. Study Site 2

3.3.2.1. Study site description

This study was carried out in Santa Eugènia's WWTP (Santa Eugènia, Mallorca, Spain). Santa Eugènia's climate is Mediterranean, with average annual rainfall of 450 mm. Fall and spring are the two rainiest seasons. Average annual temperature is 17.37 °C. Monthly average temperatures range between 10.35 °C in January and 26.37 °C in August.

The WWTP consists of a pretreatment (rotary screen) followed by a conventional secondary activated sludge treatment system, with a 180 m³ aeration chamber. The design flow is 225 m³/day, and the COD design value is 350 mg/L. The secondary settler is a rectangular clarifier with a 30 m² surface and a volume of 78 m³. The treated effluent is discharged into a pond and subsequently into a stream.

3.3.2.2. Pilot plant description

The pilot plant was constructed at Santa Eugenia WWTP. It consists of a pond feeding four parallel filters (two VFCWs and two HFCWs) (Figure 3.4.).



Figure 3.4. Pilot plant layout (Santa Eugènia)

The influent to the CWs comes from a pilot scale pond (tertiary pond). The pilot scale pond is 1.5x6x1.5 m. The pond is fed by gravity with Santa Eugenia's WWTP effluent.



Figure 3.5. View of the tertiary pond

After the pond, there are four parallel SSFCWs (two different VFCWs and two different HFCWs) with a surface area of 0.7 m^2 each. All beds were planted with *Phragmites Australis*.

<u>Vertical Flow Constructed Wetlands</u>

The VFCWs (Figure 3.6) are sequentially fed by a submerged pump controlled by an automatic valve. The filters' inlet is through a pipe which distributes the water over the entire surface. Holes 8 mm in diameter are spaced out every 250 mm along the top of each distribution pipe to disperse the wastewater over all the filter's surface.



Figure 3.6. View of the VFCWs

Two filter sand layer depths were used (70 cm and 100 cm). These VFCWs were designed with different sand depths to increase removal efficiency (mainly of microbiological indicators). Under all the beds' media there is a 10 cm transition layer of 6-10mm gravel, and at the bottom of each bed a 20 cm draining layer of 30-60mm gravel (Figure 3.7).



Figure 3.7. VFCWs media layers

The siliceous sand filtration layer has the following characteristics: $d_{10}=0.5$ mm; CU= 1.8. Sand particle size distribution curves were performed at the Soil Science laboratory of the University of Barcelona and are shown in Appendix C.

Horizontal flow constructed wetlands

The two filters are fed continuously by a dosing pump that injects the inlet water through a channel for homogeneous distribution over the entire surface (Figure 3.8)



Figure 3.8. HFCWs distribution area

The water level in the HFCWs is controlled by means of an adjustable pipe. The two HFCWs have the same area, but different gravel sizes for the treatment area (8-12 mm gravel and 18-22 mm gravel). In both filters, 40-60 mm gravel is used in the distribution (20 cm) and drainage areas (10 cm).



Figure 3.9. View of the HFCWs

Table 3.10. summarises each filter's characteristics and indicates the names used in the study to define each bed configuration.

Filter	Flow	Depth (cm)	Filtering medium	Size (mm)	CU
H1	Horizontal	60	Gravel	8-12	-
H2	Horizontal	60	Gravel	18-22	-
V1	Vertical	70	River sand	0.5	1.8
V2	Vertical	100	River sand	0.5	1.8

Table 3.10. Nomenclature and main filter characteristics (study site 2)

3.3.2.3. Experimental protocol

The pilot plant was monitored and operated for 15 months (from September 2011 to December 2012). The operating conditions and monitoring are described in the next sections.

3.3.2.3.1. Operation

HFCWs were fed continuously, and VFCWs were fed intermittently: 3.5 days of feeding and 3.5 days of resting in order to avoid clogging. Filters were fed for three minutes every two hours (12 batches/day). Different operation strategies were studied by applying different HLs. A summary of the operation periods can be seen in Table 3.11.

		Duration		Dosing	Dosing modes		
Filters	Period	(months)	n∟ (cm/day)	Batch height (cm)	Number of batches/day		
	1	8	26	Conti	nuous		
H1, H2	2	7	40	Conti	nuous		
	1	8	31	2.6	12		
VI, VZ	2	7	77	6.4	12		

 Table 3.11. Operation periods (study site 2)

3.3.2.3.2. Monitoring

Water quality monitoring

<u>Physicochemical analyses.</u> Physicochemical parameters were evaluated at

 (a) the pond outlet and (b) the filter outlet. A monitoring program consisting
 of grab sample analysis (twice a week) was implemented for 15 months. pH,
 EC, COD, dCOD, SS, Turbidity, TN, N-NH₄⁺, N-NO₃⁻, and TP were analyzed
 following Standard methods (APHA- AWWA-WPCF, 2005) or with Hanna

kits. The analytical method's references are listed in annex Appendix A. Particle-size distribution of the suspended solids in the water samples filter inlet and outlet) was determined by using a laser particle analser. Analyses were performed at the Aqualogy Laboratory in Santa Maria (Mallorca) or the Soil Sience laboratory of the University of Barcelona.

- <u>Microbiological analyses.</u> Microbiological parameters were evaluated at (a) pond outlet and (b) filters outlet. A monitoring program consisting of grab sample analysis (twice a week) was implemented for 15 months. *E. coli*, analyses were performed within 24 h, following Standard Methods (APHA-AWWA-WPCF, 2005). References are listed in Appendix A.
- <u>Algae monitoring.</u> The quantitative and qualitative algal biomass evolution was studied at the (a) pond and (b) filter outlet by optical microscopy and Chl-a analysis (AFNOR, 2005). The method used for Chl-a determination and algal identification are shown in Appendix A.

Hydraulic monitoring:

Inlet flows were determined by calculating the pump operation time.

3.3.2.3.3. Statistical analysis

Statistical analysis was performed on raw data using the statistics computer packages Excel 2010 and SPSS 16.0 for Windows. Excel 2010 was used for descriptive statistics (i.e., averages, SD). SPSS 14.0 was used for variance (ANOVA) and regression analysis. One level of significance (p) was established (p \leq 0.05). Data for these test parameters that were not normally distributed were log-transformed to present the normal distribution required to run these analyses.

3.4. Results and discussion

3.4.1. Study site 1

3.4.1.1. Facultative pond: effluent quality

The average pollutant concentrations in the pond effluent and removal efficiency of the pond are presented in Table 3.12. The quality of the pond effluent is representative of facultative ponds in France (Racault *et al.*, 1995). The mean concentrations reached by the pond did not respect the French quality level D4 for discharge in sensisitve water bodies (<125 mg/L COD, < 25 mg/L BOD₅).

 Table 3.12. Average concentration (mg/L) and SD of pollutants in the pond effluent and removal efficiency in the facultative pond (%)

Baramotor	Effluent con	Removal	
	Average	SD	efficiency
COD (mg/L)	140	45	89 %
dCOD (mg/L)	93	56	65 %
BOD₅ (mg/L)	60	34	69 %
SS (mg/L)	44	22	76 %
TKN (mg/L)	19	5.4	70 %
$N-NH_4^+$ (mg/L)	13	5.2	72 %
N-NO ₃ ⁻	< 0.5	-	-
TP (mg/L)	3.6	0.5	62 %
FC (Ulog)	4.7	0.9	2.5 Ulog
E. coli (Ulog)	4.4	0.9	2.7 Ulog

The pond performed consistently for the removal of all physicochemical and microbiological parameters. Organic matter elimination was high, especially in winter due to the low concentration of algal cells in the pond effluent. To maintain facultative conditions there must be an algal community in the surface layer (Abis and Mara, 2003) and according to Pearson *et al.* (1987) 300 μ /L Chl-a are required to guarantee stable facultative conditions. However, the removal rates of COD, BOD₅ and dCOD were very high in cold periods although the Chl-a concentration was below 100 (Figure 3.10). Removal of nutrients (70% TKN, 72% N-NH₄⁺, 62% TP) and bacterial indicators (≥ 2.5 Ulog) was fairly high.

Nevertheless, the physicochemical and microbiological quality of the final pond effluent was not suitable for discharging it in sensitive receiving bodies or for reuse which confirmed the need of an additional treatment. Fluctuations in the effluent quality according to the season were observed (Figures 3.10 and 3.11). COD, SS and dCOD were higher during the warmer periods and are related to the measured biomass (Figure 3.12). The chl-a evolution and the effluent's SS and COD trends suggest that the value of these parameters is greatly influenced by the algal cells presence. The high concentrations of dCOD are probably resulting from the excretion of carbohydrates by the algae.



Figure 3.10. Evolution of COD, dCOD and SS content in the pond effluent

On the other hand, a higher reduction of nitrogen forms and bacterial indicators in warm periods was observed. In summer, ammonia concentrations remained very often under 10 mg/L (Figure 3.11). The data suggest that ammonia removal also has a seasonal behavior: the improved ammonia elimination in summer was linked with the higher water temperatures and Chl-a concentrations. These results are according to the indications of Shilton (2006).



Figure 3.11. Evolution of N-NH4⁺ content in the pond effluent

The removal of FC ad *E. coli* was also higher in summer (> 3 Ulog) when the concentrations of chl-a were important. High ambient temperature, solar radiation and pH due to the growth of algae have been reported to encourage pathogen inactivation and die-off (Davies-Colley *et al.*, 1999; Alcalde *et al.*, 2005). The evolution of algal biomass and the above-mentioned parameters was the same for the two years of study. The only exception was the presence of the anomopod branchiopod *Daphnia* spp. that caused a decline in chlorophyll content (Figure 3.12).



Figure 3.12. Evolution of Chl-a in the pond effluent and the predominant algae genera

Daphnia spp., are mainly indistinctive filter feeders: the intensive biological filtration prevents algae to grow (Kampf *et al.*, 2007). Therefore, during that period the pond stayed clear with low algae numbers, because of the feeding behaviour of *Daphnia*. Chl-a maximum was 1518 μ g/L and minimum was 2 μ g/L. The mean Chl-a was 433 μ g/L, which is typical for a healthy facultative pond (Kaya *et al.*, 2007). The predominant algae genera are shown in Figure 3.13.



Figure 3.13. Predominant algae genera (Scenedesmus sp., Chlorella sp., Euglena sp.)

The contribution (ratio) of Chl-a to SS, COD, BOD₅ and SS was the following: 100µg/L Chl-a \approx 30 mg COD, 100µg/L Chl-a/ \approx 12 mg BOD₅, 100µg/L Chl-a/ \approx 10 mg SS, which is consistent with values observed by Pearson and König (1986) and Shiping *et al.* (2007).

The particle size counting method (laser granulometry) was used to characterise the pond effluent. Figure 3.14 shows SS particle numbers in correlation with particle size for the pond effluent in spring (May) and autumn (September).



Figure 3.14. Particle count analysis in the pond effluent in May and September

This figure clearly shows higher number of 20-30 µm-particles (that corresponds to the *Euglena* sp. size) in September. In May, the dominating genera was *Chlorella* sp., corresponding to particles around 2-10 µm. The figure also shows the increase on the total number of particles during the warmer months (with the increase of temperature and solar radiation). Air temperature and solar irradiance monitoring suggests that chl-a concentration in facultative ponds is initially affected by light and temperature. Next figures show fluctuations in Chl-a (monthly averages) with changes in air temperature and in solar radiation.



Figure 3.15. Chl-a/Air temperature (monthly averages)



Figure 3.16. Chl-a/Solar Irradiance (monthly averages)

Therefore, solar radiation is a main factor in algae development and will have a direct effect on pond effluent quality. Figure 3.17 shows particle size distribution curves in the

pond effluent in December for three samples (one grab sample taken in the morning, one at night and one a 24-hour composite sample).



Figure 3.17. Particle count analysis of pond effluent in three samples (December)

The three profiles are similar in winter periods when there is not so much algae concentration and stratification. Figure 3.18 shows particle size distribution curves in the pond effluent in June for 3 samples (one grab sample taken in the morning, one at night and one a 24-hour composite sample). The three profiles are completely different in summer periods.



Figure.3.18. Particle count analysis of pond effluent in three samples (June)

Figure 3.18 also shows a high number of particles of approximately 20-40 μ m (identified as *Euglena* spp.). The number of these particles (algae) in the morning was higher than at night. The composite sample shows an average value between the two grab samples. Solar radiation and thermal stratification in the pond result on different algae concentration in the same day and in the pond water column, and thus in the

pond outlet. Placement of the pond outlet (distance from the water surface) is of great importance. All these parameters have an important effect on algae concentration in the pond effluent and, therefore, on effluent quality.

3.4.1.2. Performance of vertical flow constructed wetland pilots

3.4.1.2.1. Pilots efficiency: effluent quality and pollutants removal

Different organic and HLs were applied along the study (20-80 cm/day; 20-170 $gCOD/m^2$ ·day during feeding periods). During monitoring, all filters achieved the water quality objectives (<125mg/L COD, <25mg/L BOD₅) based on the standards fixed by the 1997 French regulations for discharge in sensitive areas with effluent concentrations lower than 100 mg/L for COD and 20 mg/L for BOD₅ (Table 3.13).

Filtor	M6	5	R65		Ce	C65		M25		R25		C25	
Filler -	mg/L	%	mg/L	%	mg/L	%	mg/L	%	mg/L	%	mg/L	%	
	57.7	62	59.3	57	76.4	40	79.0	11	79.8	10	96.9	25	
COD	(15)	02	(18)	57	(19)	49	(16)	44	(18)	42	(17)	30	
	45.0	52	49.1	47	67.1	27	53.8	10	55.3	25	69.2	22	
UCOD	(19)	52	(21)	47	(18)	21	(16)	42	(17)	35	(15)	23	
BOD-	6.2	80	7.9	96	17.0	70	13.5	76	13.6	76	18.1	69	
BOD5	(2.5)	09	(3.5)	00	(4.1)	70	(4.2)	70	(5.7)	70	(6.2)	00	
22	9.8	78	11.5	75	19.7	60	17.1	63	17.8	60	26.3	52	
55	(6.3)	70	(5.9)	75	(11)	03	(8.6)	03	(8.2)	03	(10)	52	
TKN	4.9	78	4.3 (3.9)	4.3	6.7	70	6.9	60	6.5	70	8.7	63	
	(4.1)	70		15	(3.9)	70	(5.4)	09	(4.1)	10	(5.5)	03	
NI-NIH .+	1.7	02	1.6	02	4.0	73	3.0	82	2.7	83	4.3	71	
IN-INI 14	(3.7)	92	(2.6)	52	(3.1)	75	(4.3)	02	(4.2)	05	(4.5)	71	
	10.4	*	14.3	*	11.5	*	11.1	*	14.3	*	11.3	*	
	(6.3)		(11)		(6.4)		(4.8)		(8.8)		(7.8)		
тр	1.9	52	3.0	35	3.3	10	2.8	27	3.0	٥	3.4	2	
TP	(1.2)	52	(1.2)	30	(0.5)	10	(1.1)	21	(0.8)	9	(0.3)	2	

 Table 3.13. VFCWs performance (physicochemical parameters): average outlet pollutant concentration (SD) and % removal

* Inlet N-NO₃⁻ concentrations <0.5 mg/L

All filters were effective at removing SS (59-78% depending on the design) confirming their capability for retaining algae. The filters nitrified the facultative pond effluent, even during colder periods, with TKN concentrations < 8mg/L and $N-NH_4^+$ concentrations < 4mg/L. The TP and $P-PO_4^{3-}$ outlet concentrations ranged from 1.9 to 3.4 mg/L and from 0.1 to 2.8 mg/L, respectively. Retention of phosphorus was low; after a year of

operation the removal efficiency diminished drastically in all beds (from 90% to 20% for planted filters and from 80% to <5% for unplanted filters) (Figure 3.19). A significant retention would require the installation of specific materials (Molle, 2003).



Figure.3.19. Phosphorus removal evolution

Although all filters performed efficiently during the two years of monitoring, differences in the performances were evident depending on the studied configurations and conditions. Regarding microbiological parameters mean microbial densities (log CFU/100mL for bacteria and log PFU/100mL for phages) in the influent and effluent of the filters and removal efficiencies are shown in Table 3.14 and Figure 3.20.

						-						
Indicator	Μ	65	R	65	С	65	Μ	25	R	25	C	25
indicator	I	Е	I	Е	I	Е	I	Е	I	Е	I	Е
Fecal	4.7	3.2	4.7	3.1	4.7	3.1	4.7	4.0	4.7	4.0	4.7	4.1
coliform	(0.9)	(1.2)	(0.8)	(1.2)	(0.9)	(1.4)	(0.9)	(0.9)	(0.8)	(1.0)	(0.9)	(1.0)
E coli	4.4	2.9	4.5	3.0	4.4	2.9	4.4	3.9	4.5	3.7	4.4	3.8
<i>E. con</i>	(0.9)	(1.1)	(0.8)	(1.2)	(0.9)	(1.3)	(0.9)	(1.0)	(0.8)	(0.9)	(0.9)	(1.0)
Somatic	4.1	2.9	4.2	2.9	4.2	3.0	4.1	3.5	4.2	3.5	4.2	3.5
coliphages	(0.8)	(1.0)	(0.9)	(0.9)	(0.8)	(1.2)	(0.8)	(0.8)	(0.9)	(0.9)	(0.8)	(0.9)
F-specific	3.6	2.9	3.5	2.8	3.5	2.7	3.6	3.2	3.5	3.3	3.5	3.2
bacteriophages	(0.4)	(0.7)	(0.4)	(0.7)	(0.4)	(0.9)	(0.4)	(0.6)	(0.4)	(0.6)	(0.4)	(0.7)
							~					

 Table 3.14. Concentrations (Log CFU/100 mL or Log PFU/100 mL) of the microbiological parameters (average and SD)

I (Influent), E (effluent)

The concentrations of the indicators of fecal contamination in the pond effluent (filter inlet) were comprised between 10^4 and 10^5 CFU or PFU per 100 mL, except for F-specific bacteriophages, which registered around 10^3 PFU/100 mL. This lower

concentration was due to the higher removal of these phages in the pond. The average concentrations of the indicators at the filter outlets were in general above 1000 CFU/100 mL. The average removal of all the indicators was less than 2 log units (Figure 3.20). The removal of FC and *E. coli* were similar, between 0.5 and 2 log units depending on the filter configuration and operating conditions.



Figure 3.20. Average removal efficiencies (Log Units)

The observed bacterial indicators removals were consistent with the results obtained with other VFCWs operating at a similar HL. At HL of 50-130 cm/d, a removal of 0.7-1.5 orders of magnitude of total coliforms and fecal streptococci was observed by Arias et al., (2003). Vacca et al. (2005) showed that the number of coliform bacteria was reduced by two orders of magnitude in VFCWs at lower HL (6 cm/day). Sleytr et al., (2007) reported high removal rates for indicator organisms (4.3 log units for E. coli, 4.3 for total coliforms and 4.8 for enterococci) in a VFCW that treats a primary effluent. However, the HLs applied in the latter study were much lower: from 6 cm/day (4 feeding sequences per day) to 24 cm/day (8 feeding sequences per day) and the inlet concentrations were much higher than those applied in this study. The removal of somatic coliphages and F-specific bacteriophages ranged from 0.4 to 1.5 logs and from 1 to 0.2, respectively. The log reduction of viral indicators never reached the level observed for most of the bacterial indicators. Viral indicators (especially F-specific bacteriophages) were more resistant than bacterial ones in both planted and unplanted filters. This result confirms the findings by Campos (1998) and Folch (1999) that the removal mechanisms of viral and bacterial indicators during infiltration in porous media are diverse.

3.4.1.2.2. Algae removal

Removal of Chl-a in the filters' inlet and outlet is shown in Table 3.15.

	M65 R65		5 C65		M25		R25		C25			
	mg/L	%	mg/L	%	mg/L	%	mg/L	%	mg/L	%	mg/L	%
	68	00	76	04	116	74	172	F7	192	50	208	40
Chi-a	(21)	83	(419)	81	(17)	/1	(25)	57	(19)	52	(34)	48

Table 3.15. Chl-a removal average outlet pollutant concentration (SD) and % removal

Removal of Chl-a showed the same pattern as SS and BOD₅ with similar percentage removal of SS (Chl-a removals between 48-83 % depending on design and operation). These data confirm the filters' capability for retaining algae. Algae retention is much higher in the deeper filters, both planted and unplanted, and slightly worse for crushed sand. Figure 3.21 shows algae in the 25cm-depth filter effluent.



Figure 3.21. Algae in the R25 filter outlet

These percentage removals in SSFCW are higher than those reported in the literature (Table 3.4). The percentage of removal for the three filters of 65 cm depth was about 70-80 %. When the filters presented a deposit on the surface, the algae removal was even higher (> 80%).

3.4.1.2.3. Hydraulics

The nominal HL in the filters was 250 L/PE (75 cm/day). The established feeding and resting periods were 3 to 4 days of feeding, followed by a 7 day rest period. Filters were intermittently fed: flooding sequences alternating with drainage sequences. The number of daily feeding-drainage cycles (f) depended on the HL applied and the dose. Table 3.16 presents a summary of the loads and operating conditions.

	D	Hydraulic	Average	Dosing modes				
Stage	Duration (months)	Load (HL) (cm/day)	Organic - Load (COD/m²⋅day)	Height of batch (cm)	Number of batches per day (f)			
1	3	20-25-	17-21	5	4-5			
2	10	35-40	42-68	5	7-8			
3	5	75-80	104-140	5	15-16			
4	5	75-80	119-170	2.5	30-32			

Table 3.16. Summary of loads and operation parameters

Tracer tests

Tracer tests made it possible to calculate the mean HRT and the DTD curves. The summary of the tracer test conditions and the calculated mean HRT for each test are shown in Table 3.17.

Tracer	Filtoro	HL	Bactch	Number of	Mean HRT
test	riiters	(cm/day)	height	batches per day	(hours)
1	R65	25	E om	7	10.0
1	R25*	35	5 CIII	/	10.4
2	C65	25	E om	7	11.1
Z	C25	35	5 011	/	5.1
2	R65	40	5 om	0	7.4
3	R25	40	5 011	o	4.1
Л	M65	75	5 cm	15	11.1
4	M25*	75	5 611	15	11.4
Б	R65	75	5 cm	15	6.2
5	R25	75	5 611	15	3.3
6	C65	40	5 cm	8	8.4
0	C25	40	5 611	0	6.2
7	M65	40	5 cm	8	6.6
,	M25	40	5 611	0	2.3
8	R65	40	5 cm	8	7.1
0	R25	40	5 611	0	4.1
0	R65	75	2.5 cm	20	6.9
J	R25	75	2.0 011	50	3.2
10	C25	75	2.5 cm	30	2.8
11	R25	75	2.5 cm	30	3.4

Table 3.17. Tracer tests conditions and results: mean HRT

*Filter with a deposit on the surface

The mean HRT varied from 2.3 hours to 11.4, depending on the filters' design and operation. The effect of the different design, operational parameters and the surface algae on the mean HRT and the DTD curves are shown in next sections.

Infiltration rates

Constant monitoring of infiltration rates was a practical tool to study the filters' hydraulic performance. In general, filters showed good infiltration, with general values $>1.10^{-4}$ m/s (without not deposit on the surface). For all filters, infiltration rates decreased progressively with every succesive batch (feeding). It is worth noticing that after a certain number of feedings (15 batches, approximately) infiltration rates stopped evolving and became stable. Figure 3.22 shows infiltration rates for a constant rate feeding cycle for Filter 1-River Sand 65 cm (HL0 20 cm/day and one batch every 4.5 hours). This trend was observed in all filters, as in Molle's (2003) experiments with VFCWs. Fast infiltration at the very beginning of each feeding period may be related to the high pressure differences due to low humidity conditions inside the filter beds. While filters are fed and become humid, infiltration rates decrease. At the end of the cycle, infiltration rates become stable.



Figure 3.22. Evolution of infiltration rates during a feeding cycle in the R65 filter (after 6 days of resting)

Table 3.18 shows the orders of magnitude of the IRs for different filters, and the corresponding value is recorded under "Stabilization".

	IR average v	alues (m/s)	
Filter	First batch	Batch 10	«Stabilisation»
R65	1 - 1.5.10 ⁻⁴	2 - 6.10 ⁻⁵	2 - 5.10 ⁻⁵
R25	>1.10 ^{-4.}	8.1 - 5.10 ⁻⁴	2 - 7.10 ⁻⁵
C65	2 - 6.10 ⁻⁵	7.10 ⁻⁶ - 10 ⁻⁵	2 - 5.10 ⁻⁶
C25	>1.10 ^{-4.}	4 - 6.10 ⁻⁵	4 - 6.10 ⁻⁵
M65	4.10 ⁻⁶ - 2.10 ⁻⁵	3.10 − 6.10 ⁻⁵	4 - 6.10 ⁻⁶
M25	> 1.10 ^{-4.}	4 - 9.10 ⁻⁶	4 - 6.10 ⁻⁶

 Table 3.18. Magnitude of IRs in the filters (first year of operation: HL=20-40 cm/day)

The second year in operation, with no clogging layer and HL of 75-80 cm/day, all filters almost always showed values $>1.10^{-4}$ m/s, even on the third feeding day.

3.4.1.3. Effect of design and operational parameters

3.4.1.3.1. Effect of the presence of Phragmites

Planted and unplanted beds filled with river sand presented similar infiltration rates ($\approx 1.10^{-4}$ m/s) and DTD curves (Figure 3.23). Although the DTD curves are very similar for both kinds of filter, a fraction of the water from the first batches flowed through the planted beds more quickly than through unplanted filters (possibly due to the presence of rhizomes creating preferential pathways).



Figure 3.23. DTD curves for planted and unplanted filters of 65 cm (HL=40 cm/day, f=8)

Planted filters performed slightly better for removal of organic matter, TKN and N-NH₄⁺ (Table 3.19), but these differences were not statistically significant (p>0.05). This suggests that ammonia assimilation by plants, as well as their role in rhyzosphere oxygenation, is of minor importance in vertical filters, which is in agreement with Reed *et al.*, (1995) and Keffala and Ghrabi (2005).

	CO) BO		D ₅ SS		6	TKN		N-NH₄ ⁺		N-NO ₃ ⁻	
	mg/L	%	mg/L	%	mg/L	%	mg/L	%	mg/L	%	mg/L	%
Mee	57.7	62	6.2	80	9.8	79	4.3	70	1.6	02	10.4	(*)
IVIO5	(15)	02	(2.5)	09	(6.3)	10	(3.9)	19	(2.6)	93	(6.3)	()
DGE	59.3	57	7.9	05	11.5	75	4.9	70	1.7	00	18.5	(*)
R05	(18)	57	(3.5)	60	(5.9)	75	(4.1)	10	(3.7)	90	(11)	()
MOE	79.0	11	13.5	76	17.1	62	6.9	70	2.7	02	11.1	(*)
10125	(16)	44	(4.2)	70	(8.6)	03	(5.4)	70	(4.2)	63	(4.8)	
P25	79.8	12	13.6	76	17.8	62	6.5	60	3.0	82	14.3	(*)
R25	(18)	42	(5.7)	10	(8.2)	03	(4.1)	09	(4.3)		(8.8)	

 Table 3.19. Performance of planted and unplanted filters: average outlet pollutant concentration (SD) and % removal

^{*} Inlet N-NO₃⁻ concentrations <0.5 mg/L

Nevertheless, the average reduction in $N-NH_4^+$ during colder periods in planted filters was significantly higher (*p*<0.05) than in the unplanted ones (Figure 3.24).



Figure 3.24. N-NH₄⁺ removal in winter for planted (M65, M25) and unplanted (R65, R25) filters

Two major parameters that affect microbial nitrification are oxygen availability and temperature. The reduction-oxidation curves for the filters effluents were in aerobic conditions and did not show any difference between beds. Hence, the differences in N-NH₄ removal performance could be partially explained by the different temperatures inside the planted and unplanted filters (Figure 3.25).



Figure 3.25. Temperature at 15 cm inside for planted (M65) and unplanted (R65) filters

As noticed by Brix (1994), air temperature variations are attenuated in the planted filters due to the vegetation cover and therefore the wastewater temperature inside the filters with macrophytes is warmer during the colder periods than in the unplanted ones. Figure 3.15 shows that temperature of planted filters was more constant and about 4°C higher during the night periods tan the unplanted ones. As nitrification activity is dependent on temperature, planted filters would have better nitrification performance and therefore a higher N-NH₄⁺ removal efficiency tan the unplanted filters.

None of the filters effectively removed TP or $P-PO_4^{-3-}$ (<5% for unplanted and around 20% for planted filters during the second year of operation). The statistically significant better performance (*p*<0.05) of the planted filters was probably due to the assimilation of phosphorous by the plants. Regarding the maintenance of the filters, when working with planted beds, at the beginning of winter every year the faded aerial part of the reeds was cut and removed from the beds. Unplanted filters did not need this maintenance activity but their maintenance was more frequent and complicated due to the continuous growth of weeds that had to be removed every week. These weeds did not grow in the planted beds due to the presence of *Phragmites*.

Regarding microbiological indicators, no significant difference in the removal efficiency of planted and unplanted vertical flow beds was observed (p>0.05). The overall removal efficiency of the filter plots for all the microbiological indicators was similar for filters of the same depth, regardless of whether the plots were planted or not, as has been shown in Figure 3.20. These results indicate that the presence of macrophytes is of minor importance for the removal of microorganisms in vertical filters intermittently dosed, which is in agreement with Sleytr *et al.* (2007) and Vacca *et al.* (2005). The tracer tests showed that planted and unplanted filters presented the same flow patterns. Moreover, the oxygenation conditions in planted and unplanted filters were similar. In intermittent sand filtration oxygen transfer into the media is mainly achieved by convection due to batch loading and diffusion processes from the air. Even though the oxygen provided by this plant in relation with the other sources of oxygen is negligible in vertical filters intermittently dosed.

3.4.1.3.2. Effect of the depth of the filter

Tracer results showed that water flowed through the 25-cm filters faster than through the 65-cm filters (Figure 3.26).



Figure 3.26. DTD curves for R65 and R25 (HL = 40 cm/day, f = 8)

With the same feeding regime for each kind of media, the 25-cm filters had a HRT lower than the 65-cm filters. Mean HRTs ranged from 1 to 5 h for the 25 cm filters and from 4 to 11 h for the 65 cm filters. A depth increase of only 40 cm resulted in an augmentation of the mean HRTs from 3 to 6 h. The dose applied in every feeding sequence allowed saturating the 15 cm top layer of the filters. Therefore, taking the existence of a capillary fringe into account, water pulses infiltrated through saturated or nearly saturated 25 cm deep filter beds. Water saturation assured a maximum hydraulic conductivity and high water velocities. The same dose was too small for the water content being close to saturation throughout the 65-cm deep filters. As hydraulic conductivity decreases very rapidly with the water content, water transferred more slowly in the low half of 65-cm deep filters.

The 65-cm filters performed significantly better (p<0.05) than 25-cm filters with respect to all the physicochemical parameters, particularly with respect to the removal of organic matter (Table 3.19. and 3.20).

Ellis	COD	COD		dCOD		BOD₅		SS			N-NH₄ ⁺		
Fliter	mg/L	%	mg/L	%	mg/L	%	mg/L	%	mg/L	%	mg/L	%	
CGE	76.4	40	67.1	27	17.0	70	19.7	60	6.7	70	4.0	72	
005	(18)	49	(18)	21	(4.1)	70	(11)	09	(3.9)	70	(3.1)	13	
Dee	59.3	57	49.1	47	7.9	96	11.5	75	4.9	70	1.7	92	
ROD	(18)	57	(21)	47	(3.5)	00	(5.9)	75	(4.1)	10	(3.7)		
0.05	96.9	25	69.2	22	18.1	60	26.3	50	8.7	60	4.3	74	
625	(17)	35	(15)	23	3 (6.2)	00	(10)	52	(5.5)	03	(4.5)	71	
DOF	79.8	40	55.3	25	13.6	76	17.8	60	6.5	60	2.7	00	
R20	(18) 42	42	(17)	30	(5.7) ⁷⁶		(8.2)	63	(4.1)	69	(4.2)	02	

Table 3.20. Performance of unplanted crushed sand and river sand filters: average outlet pollutant concentration (SD) and % removal

The short HRT of the shallow beds probably did not allow increased removal. The effluent from the 25 cm filters had COD and BOD₅ concentrations close to the fixed

discharge limits (<25 mg/L BOD₅, <125 mg/L COD). Filter depth also had a significant effect (p<0.05) on algae removal, as demonstrated by the granulo-laser analysis and SS removal. Microscopic and biological analyses of the filter sand showed that algae retention, especially in the case of round and small algae (e.g. *Chlorella* sp.), not only occurs at the surface of the filter but throughout the medium.

The filters with a depth of 65 cm presented significant (p<0.05) higher removal of bacterial and viral indicators than those of 25 cm (Figure 3.10). The short HRTs of the shallow beds (less than 3 hours in general) may not allow noticeable disinfection.

3.4.1.3.3. Effect of different sand types

The IRs and the tracer tests lead to no consistent conclusions concerning the hydraulic behaviour of the different types of sand. Additional tests must be performed in order to compare the hydraulic patterns of the two media. Statistical analysis of filter performance (Table 3.20) indicated that the type of sand had a significant effect on the removal of all pollutants. Crushed sand filters performed significantly worse (p<0.05) than river sand filters in all the conditions tested. These results could be partially explained by the shape of the crushed sand (more angular). Analysis of the biomass in the filters demonstrated that the biomass content was lower in the crushed sand filters (*Torrens et al.*, 2009a). The angular form and roughness of the crushed sand would make the attachment of the biomass more difficult. Moreover, the heterogeneous compaction of the crushed sand, would lead to heterogeneous pore-size distribution, thus creating preferential pathways that could explain the lower removal efficiencies of crushed sand filters.

No significant difference (p>0.05) was either found between crushed and river sands for any microbial indicator. These results indicate that if the particle size distribution is suitable, the use of alluvial material is not indispensable regarding the removal of microbial indicators. Sand from quarries may also be used after crushing and sieving.

3.4.1.3.4. Effect of the operational parameters: hydraulic load and dosing regime

In general, the increase HL reduced the removal efficiency of the filters: an important decrease on COD, SS and TKN removals were observed when doubling the HL (Figure 3.27).



Figure 3.27. COD, SS and TKN removal for hydraulic loads of 80 cm/day and 40 cm/day

High HLs resulted in a reduction in disinfection capacity. Fig. 3.28 presents the average log removal of the microbiological indicators for HL around 20 cm/day, 40 cm/day and 80 cm/day. The increase of the HL significantly decreased the removal rates for FC, *E. coli* and somatic coliphage indicators (p<0.05). Tracer tests showed that the detention time decreased with increased HL. Lower purification, as a result of increased dosing rate, has also been observed in several experiments with intermittent infiltration in porous media (Salgot *et al., 1996;* Brissaud *et al., 1999)*. However, the removal of F-specific bacteriophages was not significantly affected by the HL (p>0.05). The log removals of this viral indicator were always lower than 1 Ulog irrespective of the HL applied.



Figure 3.28. Removal of microbiological indicators for a range of HLs

Regarding dosing regime, DTD was strongly influenced by the fractioning of the daily hydraulic load (f) in agreement with the observations of Brissaud (1999) and Molle

(2003). For the same HL (75cm/day), when f was lower (i.e. higher dose volume), a great part of the applied water quickly passed through the bed. Tracer breakthrough occurred for less than 1 h when f=15 (Figure 3.29).



Figure 3.29. DTD curves of R65 for f=15 and f=30 (HL=75 cm/day)

When f=30 (i.e. lower dose volume) tracer breakthrough occurred after 2h. A high dosing rate increases the water velocity through larger pores and reduces the exchange between mobile and less mobile water. Hence, by decreasing the batch volume, HRT is increased and there is greater exchange of the less mobile fraction of the pore water. This circumstance will allow closer and longer contact between media and pollutants (Stevik *et al.*, 2004).

The dosing regime also played an important role in determining the level of treatment in the filter (Table 3.21). During the first days of feeding, performances of reduction for COD, SS and TKN were significantly better (p<0.05) at higher *f*-values for all the 65-cm filters. These results are in agreement with Folch (1999) and Molle *et al.* (2006) results. COD removal and oxidation of nitrogen appeared to be highly dependent on f. Folch (1999) and Bancolé *et al.* (2004) showed that the more the daily load is divided, the greater the removal of organic matter and nitrogen. Nevertheless, batch frequency has different impacts on the evolution of COD removal and nitrification efficiency.

Parameter	C	DD	S	S	TKN		
Filter	f=15-16	f=30-32	f=15-16	f=30-32	f=15-16	f=30-32	
C25	33.0	34.6	41.8	44.4	65.7	70.6	
R25	24.9	27.0	46.1	49.0	71.2	78.7	
M25	33.3	38.2	51.1	52.0	71.0	78.8	
C65	45.9	53.8	63.6	66.0	64.0	77.1	
R65	40.8	58.7	66.5	81.8	68.2	87.3	
M65	42.9	60.9	68.0	82.2	72.0	88.4	

Table 3.21. Removal efficiency (%) over the first 2 days of feeding (HL=75-80 cm/day)

While COD removal remained constant over time when f was 30-32, nitrification decreased further (Table 3.22) as found by Molle *et al.* (2006). Nitrification is optimized during periods of time between batches. Then, when the fractionation is higher HRT is large; however, oxygenation is low due to a decrease of oxygen diffusion into the system. Moreover, Bancolé *et al.* (2004) showed that when using low fractioning of doses per day, biofilm develops evenly over the whole depth of the filter bed, but it accumulates in the upper layers at high fractioning. As the biomass increases at the surface the hydraulic conductivity diminishes, reducing infiltration velocities and impeding the oxygen diffusion potential. When *f*=30-32, a larger dosing period than that established (3-4 days) could limit the oxygenation and the nitrification capacity, especially for the 65-cm deep filters. Hence, the number of doses per day and the periods of dosing must be limited and adapted in each case (sand granulometry, depth of the bed, HL...) to assure the sustainability of the process.

 Table 3.22. Nitrification (%) on the first and fourth day of feeding for the 65-cm filters (HL=75-80 cm/day).

Filter	R65					С	65		M65			
	f=1	5,16	f=3	f=30,32		l5,16 f=30,32		0,32	f=15,16		f=30,32	
Day of feeding	1 st	4 th										
Nitrification (%)	68	69	88	67	64	60	77	58	73	71	87	64

The results obtained for the fractionation (f=15-16, f=30-32) of the total HL (80 cm/day) showed that the high fractionation (lower water height per unit dose) significantly increased the removal efficiency for FC, *E. coli* and somatic coliphages (p<0.05) (Figure 3.30).



Figure 3.30. Removal of microbiological indicators for a range of batch frequencies (f)

Previous studies in infiltration-percolation systems also demonstrated that the higher the f value, the greater the removal of fecal indicators (Brissaud *et al.*, 1999, Folch, 1999). However, the removal of F-specific bacteriophages was not significantly affected by the fractionation (p>0.05). A longer contact between media and microbial indicators and results in enhanced bacterial adsorption and higher purification. Regarding microbiological indicators, if an increased HL is required, a higher number of doses would be preferable to an increase in the volume of the batch for the removal of microorganisms.

The results indicate that the depth of the filter, the HL and the fractionation of the daily HL significantly affect microorganism removal rates. All these design and operational variables were directly related to HRT. Great depth of filters, low HLs and low volume per batch resulted in high wastewater retention time. Higher HRT would enhance the adsorption of the microorganisms, which is one of the main mechanisms of microorganism immobilization in porous media. To study the effect of the HRT on the removal of these organisms, a correlation between the log removal of FC and the mean HRT was performed. In addition, a second correlation between the removal rates and a minimum retention time was done. Although both parameters had a significant effect on the FC removal (p<0.05), a better correlation was found using the minimum retention time (Figure 3.31).


Figure 3.31. FC removal (Ulog) plotted against minimum HRT and mean HRT

The use of a minimum retention time instead of mean retention time is more appropriate for predicting microorganism removal on the basis of breakthrough curves in unsaturated filters. This minimum retention time would better represent the fraction of the dose applied that spends the least time in the vertical filters. This fast moving fraction of the flow would contribute to increased numbers of FC in the effluent, as this fraction is lead through the largest pores of the filter media where conditions are less favorable for both straining and adsorption (*Stevick et al.*, 1999).

3.4.1.4. Characterisation and effects of deposit on the surface of the vertical flow constructed wetlands

A surface clogging of both planted and unplanted filters appeared after six months of operation. Failure to respect the recommended feeding and resting periods (3-4 days feeding/7 days resting) coupled with the presence algae (*Scenedesmus* spp.) in the pond effluent enhanced the build-up of a surface organic clogging mat. The alternation of periods of rest and feeding regulates the growth of biomass and the formation of surface deposits, thus minimizing the risks of clogging. While respecting the operation mode of 3-4 days feeding and 7 days of rest for each filter, no problems of clogging appeared with hydraulic and organic loads up to 80 cm/day and 170 gCOD/m²·day, respectively. However, continuous feeding (>8 days) or too short rest periods (of the order of 3-4 days or less) leads to a weak mineralization of the surface deposit composed of algae. On the surface of the 25-cm river sand filter (R25) a 7-mm deposit was formed (Figure 3.32), and removed manually. The deposit presented an average percentage of DM of 25% and 51 % VS.



Figure 3.32. Deposit on the surface of the filter (R25)

Two tracer tests and performance analysis (HL 20 cm/day) were carried out on the R25 filter; once with the deposit present and a second time after the deposit had been removed. Hydraulic behaviour and treatment performance in the two cases were markedly different despite the limited depth of the deposit. The outflow was much faster after the clogging layer had been removed and thus less water was remaining in the filter, as reflected in the DTD curves (Figure 3.33).



Figure 3.33. DTD curves of R25 for clogged and unclogged conditions

The mean HDT was much shorter when there was no algae deposit (about 10h with surface deposit and about 3h after the surface deposit had been removed). This was also demonstrated by the continuous IR monitoring. The IR values were very different: between 10^{-6} and 10^{-7} m/s with the deposit presence and greater than 1.5×10^{-4} m/s after the surface deposit had been removed. Clogging by algae greatly decreased IR because almost all the algae and SS were filtered at the surface if the particles (algae) were bigger than 20-30 µm (Figure 3.34).



Figure 3.34. Particle count analysis at the inlet and outlet of the R25 for clogged and unclogged conditions

Standing water at the surface hindered oxygenation, creating an anoxic state (Figure 3.35). Moreover, standing water favoured rapid algae development and increased clogging. Controlling the feeding and resting periods is thus of great importance for the durability and the reliability of the system.



Figure 3.35. Redox conditions for R25 for clogged and unclogged conditions

The maintenance of the filters was simple but needed to be regular. If surface clogging appears, the removal of this surface layer is necessary to restore the infiltration capacity. However, for planted filters the manual elimination of this layer is not possible. In that case specific hydraulic loads have to be applied until the clogging disappears by itself. Altthough this inconvenience for planted filters, the maintenance operations during the study were more frequent and complicated in the unplanted beds due to the continuous growing of weeds.

3.4.2. Study Site 2

3.4.2.1. Tertiary pond: effluent quality

The average, maximum and minimum pollutant concentrations of pond effluent are shown in table 3.23.

	Average	Min	Max
EC (mS/cm)	1.7	1.1	1.9
рН	8.0	7.1	8.8
SS (mg/L)	20.1	5	109
Turbidity (NTU)	31.8	1.4	148
COD (mg/L)	78.2	12.1	364
dCOD (mg/L)	59.2	264	0.6
TN (mg/L)	12.4	1.2	25
N-NH4 ⁺ (mg/L)	9.4	2	21
N-NO ₃ ⁻ (mg/L)	1.6	0.2	6.7
TP (mg/L)	2.9	0.5	10.5
E. coli Ulog (CFU/100ml)	5.5	6.4	3.1

 Table 3.23. Tertiary pond effluent quality

The concentration of algae and associated parameters was not so high compared to the facultative ponds of case study 1. Figures 3.36 and 3.37 show the evolution of SS, COD and Chl-a.



Figure 3.36. Evolution of COD and SS content in the pond effluent



Figure 3.37. Evolution of Chl-a in the pond and the predominant algae genera.

Figures 3.36 and 3.37 clearly show the direct relationship between algae (Chl-a) and COD and SS. The Chl-a increase took place between May and June, when temperatures and solar radiation were higher. However, in general, the average concentration of algae and associated parameters was low (average SS, average COD and average Chl-a)

Maturation or tertiary ponds after facultative ponds are mainly designed to provide favorable conditions for algal growth (i.e., shallow ponds, low organic loadings and long retention times) (Valero and Mara, 2009). However, algal concentration in polishing (tertiary ponds after conventional systems) can vary widely depending on the previous treatment. In this case study, low nutrient concentrations in the inlet of the tertiary pond together with the short pond HRTs (from 10 to 20 days), did not allow greater algae growth in the pond.

Chl-a maximum was of 726 μ g/L, and minimum of 2 μ g/L. with an average of 211 μ g/L, which is a low value for ponds. The ratio of Chl-a to COD and SS found in this study was: 100 μ g/L Chl-a \approx 37 mg COD and 100 μ g/L Chl-a/ \approx 12 mg SS. The SS ratio values are consistent with the values found in the literature (*Kaya et. al.*, 2007). However, for COD the ratio is different: 1 μ g/L Chl-a corresponded to a higher COD values. This is because a big part of the organic matter was not algal organic matter but dissolved organic matter coming from secondary treatment, and not degraded in the pond. Figure 3.37 shows the dominant algae genera found in pond effluent.



Figure 3.38. Predominant algae genera (Chlorella spp., Euglena spp.)

Laser granulometry was also used to characterize the pond effluent. Figure 3.39 shows the particle size and number of particles/100 mL in the pond effluent in June. The figure clearly shows a higher number of particles, approximately 2-10 μ m (corresponding to *Chlorella* spp.) and 20-40 μ m (corresponding to *Euglena* spp.).



Figure 3.39. Particle count analysis in the pond effluent

3.4.2.2. Performance of vertical and horizontal subsurface flow constructed wetland pilots

3.4.2.2.1. Pilots efficiency: effluent quality and pollutants removal

Different HLs and organic loads and were applied throughout the study. For HFCWs, 26-40 cm/day (18-30 gCOD/m²·day and 5-6 gSS/m²·day); 31-77 cm/day for VFCWs (23-50 gCOD/m²·day and 6-14 gSS/m²·day for filters in operation). Filters received overall low surface loading rates. These loading rates were much lower than those for VFCWs in case study 1.

Average inlet pH and EC did not present significant variations. Inlet pH was 8.1 and in filters' outlet: 7.7 (H1), 7.7 (H2), 8.1 (V1), 8.1 (V2). EC average inlet was about 1.8 mS/cm in the filters' inlet, and it did not change in the filters' outlet: 1.8 mS/cm (H1) 1.9 mS/cm (H2), 1.8 mS/cm (V1), 1.8 mS/cm (V2). The average inlet and outlet concentration (COD, SS and turbidity) and percentage removal are shown in Table 3.24. In general, HFCWs were more efficient than VFCWs for SS and turbidity removal. No significant differences were found regarding organic matter, with removals between 40-50%.

Filters		COD			SS		Т	urbidity	
	Inlet	Outlet	%	Inlet	Outlet	%	Inlet	Outlet	%
H1	78.6±63	37.3±29	47	20.3±25	3.5±3	71	33.6±29	10.7±8	61
H2	78.6±63	42.7±30	45	20.3±25	6.6±7	58	33.6±29	15.5±15	54
V1	62.4±39	41.1±20	42	24.8±26	16.0±15	28	37.7±30	24.3±24	24
V2	81.0±47	42.8±27	54	24.2±28	8.2±8	58	38.7±32	15.8±8	55

 Table 3.24.Concentrations (mg/L and NTU) and removal efficiencies for the filters (average and SD)

Removal of SS and associated algae was very high for all filters (between 67-73%) with SS concentrations higher than 40 mg/L SS (Table 3.24). For low inlet algae concentrations (SS < 20 mg/L), HFCWs are much more efficient than VFCWs. With low SS concentrations, the water percolates too fast into the filters, so HRT is short, mainly in the V1 (VFCW of 70 cm). Short HRTs do not allow biomass to properly develop inside the filters (Gallinas *et al.*, 2013). Therefore, with low algae concentration effluent, VFCWs need finer sand. This is different for HFCW and VFCW: HFCWs' removal efficiency was not affected much by SS concentration as much as in VFCWs.

	Average removal (%)						
Filters	SS (mg/L) influent < 20	SS (mg/L) influent 20-40	SS (mg/L) influent > 40				
H1	68	74	73				
H2	52	49	67				
V1	19	37	70				
V2	34	59	72				

Table 3.25. Average removal of SS (%) depending on influent SS concentration

Regarding nutrients (Table 3.26), differences in performance between both filters were obvious: VFCWs nitrify the influent and HFCWs do not (as it is mainly an anaerobic reactor). For ammonia, percent removals are better in VFCWs with high nitrification

efficiency. For total nitrogen, removals are better in HFCWs, as TN include the nitrates produced during VFCWs' nitrification.

Filtor		TN			TN N-NH4 ⁺				N	I-NO3 ⁻	TP		
Filter	Inlet	Outlet	%	Inlet	Outlet	%	Inlet	Outlet	%	Inlet	Outlet	%	
H1	11.1±7	3.97±3	62	9.6±6	5.0±4	48	1.5±0.5	1.5±0.5	-	2.2±0.8	1.9±0.9	9	
H2	11.1±7	6.9±4	43	9.6±6	6.4±5	35	1.5±0.5	1.5±0.5	-	2.2±0.8	2.2±0.8	-	
V1	9.6±6	9.4±7	17	9.4±5	4.3±3	53	1.6±0.7	6.6±1.2	*	2.3±0.7	2.4±4	-	
V2	12,4±7	13.3±7	18	10.3±5	4.2±3	65	1.5±0.6	5.7±0.9	*	2.2±0.8	2.2±0.8	-	

 Table 3.26. Concentrations of nutrients (mg/L) and removal efficiencies for the filters (average and SD)

Phosphorous removal was negligible. In this case, it is necessary to consider the limitations of the technique of analysis that cannot accurately measure changes for such low concentrations. Therefore, the results are difficult to interpret. However, significant phosphorus retention on CWs would require installation of specific materials (Molle, 2003; Johannesson *et al.*, 2015). Regarding microbiological parameters, the concentration and removal rates are shown in Table 3.27.

Filtor		E. COli	
	Inlet	Outlet	ULog
H1	6.6±0.1 x 10 ⁵	3.6±0.1 x 10 ³	1.7
H2	$6.6\pm0.1 \times 10^5$	$4.8\pm9.3 \times 10^3$	1.3
V1	$3.7\pm4.1 \times 10^4$	8.9±0.1 x 10 ³	0.8
V2	1.1±1.9 x 10 ⁵	$2.2\pm6.2 \times 10^4$	0.9

Table 3.27. Concentrations (CFU/100 mL) and removal (ULog) of *E. coli* (average and SD)

The removal of bacterial indicators was between 0.8 and 1.7 Ulog. For VFCWs, removals were <1 Ulog. Better performance was expected with VFCWs with 70 and 100 cm sand filters (Arias *et al.*, 2003, Torrens *et al.*, 2010). Microorganism removal during infiltration in porous media is normally attributed to the combination of filtration, adsorption and inactivation. The efficiency of these processes is related to several factors. Filtration is influenced by the physical characteristics of the filter medium, hydraulic loading and clogging. Adsorption is controlled mainly by the grain surface characteristics of the porous medium (Torrens *et al.*, 2009b). The sand's d₁₀ was bigger in this study than in case study 1, where percentage removals were higher, even though the media depth was lower. Sand particle size will affect the mean HRT, and HRT is a key factor on the removal of bacterial indicators in a VFCW intermittently

dosed VFCW. Horizontal filters obtain better percent removals than vertical filters, with a difference of 1 ULog between them.

3.4.2.2.2. Algae removal

Both filters (VFCW and HFCW) were effective to retain part of the SS and, consequently retain algae, as proved by microscopy and Chl-a analysis (Table 3.28). Particle count analysis (Figure 3.39) shows the SSFCWs' capacity for algae retention.

Filters		Chl-a	
	Inlet	Outlet	%
H1	211±67	65.2±21	69
H2	211±73	82.3±24	61
V1	217±74	143±47	34
V2	204±74	83.6±29	59

Table 3.28. Chl-a concentrations (µg/L) and removal efficiencies for filters (average and SD)

Chl-a removal ranged between 34% and 69% depending on the filter's design and operation. Percent removal and behavior was similar for SS.



Figure 3.40. Particle count analysis at the VFCWs inlet and outlet (V1, V2)

Figure 3.40 shows that the VFCWs effectively retain a percentage of particles (and thus algae), mainly the bigger particles (*Euglena* spp.). The filtering of algae in VFCWs will also depend on influent particle size (algae size). Particle analysis shows that particle removal between 2-10 μ m is about 30% for V1 and 45% for V2, and for

particles between 20-40 μ m, it is about 70% for V1 and 72% V2. Thus, the degree of filtration depends on particle size of VFCWs' influent.



Figure 3.41. Particle count analysis at the HFCWs inlet and outlet (H1, H2)

However HFCWs (Figure 3.41) do not behave the same way: for particles between 2-10 μ s, percent removal is about 70% for H1, and 65 % for H2. For 20-40 μ m-size particles, percent removal is 75% for H1 and 61 % for H2. This means that the sedimentation process is of great importance in the HFCWs. The degree of filtration did not depend on the SS particle size for HFCWS as much as for VFCWs (where the filtration mechanism is the most important SS removal mechanism).

3.4.2.3. Effect of design and operational parameters

3.4.2.3.1. Effect of the depth in vertical flow constructed wetlands

The 100-cm filters showed better removal performance for all physicochemical and microbiological parameters (Fig. 3.41), which is consistent with Torrens *et al., (*2009b). Tracer tests (Gallinas *et al., 2013*) showed that water flowing out through the filter was faster in the 70-cm filters than in the 100-cm ones. Eventhough the difference is only of 30-cm of sand, the shallow bed presented lower HRTs and this resulted in worse removal performance. Moreover, as presented in Table 3.27 and Figure 3.40, removal of Chl-a and algae was also higher in the deeper filters.



Figure 3.42. Effect of filter depth in VFCWs: % removal for COD, SS, N-NH₄⁺ and ULog removal for *E. coli*

3.4.2.3.2. Effect of the gravel size in horizontal flow constructed wetlands

Figure 3.43 shows the effect of gravel size in the percentage removal or Ulog of different parameters. The effect of gravel size on removal efficiency in HFCWs is still not clear, and some authors state that, if working with gravel between 10 and 60 mm, size does not significantly affect the performances. It is reported that the diameter size of media used in HF wetlands varies from 0.2 mm to 30 mm (Reed *et al.*, 1995; UN Habitat, 2007). However the study showed that HFCWs with finer gravel (8-12mm) had significantly (p<0.05) higher reduction for SS, N-NH₄⁺ and *E. coli*.



Figure 3.43. Effect of gravel size in HFCWs: % removal for COD, SS, N-NH₄⁺ and ULog removal for *E. coli*

3.4.2.3.3. Effect of the hydraulic load in horizontal and vertical subsurface flow constructed wetlands

For each type of filter (HFCWs or VFCWs), two HL were tested (labeled low HL and high HL). Effects of the increase of HL were similar in the two types of filters (HFCWs and VFCWs) (Figure 3.44). The HL did not significantly (p>0.05) affect removal of COD and *E. coli*. However, SS and nitrogen removal was affected (p<0.05) by the HL. As it has been shown in section 3.4.1.3., higher HL increases the water velocity through the media and thus could diminish the removal of some parameters due to the shorter HRTs on VFCWs.



Figure 3.44. Effect of HL in HFCWs and VFCWs: % removal for COD, SS, N-NH4+ and ULog removal for E. coli

Total COD was not affected by the increase of the HL (p>0.05), however particulate COD was affected, as well as SS and Chl-a. Thus, algae and organic matter related to algae was affected by HL. dCOD (more than 60% of total COD entering the filters) was not affected by the increase of the HL (p>0.05). The dCOD entering the filters was not algal-COD, but dCOD not degraded in the activated sludge and tertiary pond system.

Sasa (2014) showed that dCOD performance removals in HFCWs treating secondary effluent from activated sludge were not high (<40% removal) wherever the applied HL. For VFCWs, the behaviour in this study is similar to the HFCWs: this dCOD is not considerably removed in VFCWs and is not affected by the HL).

Regarding microbiological parameters, HRT is the key parameter for *E. coli* removal for both VFCWs and HFCWs (Garcia *et al.*, 2003; Torrens *et al.*, 2010; Sasa, 2014). However in the VFCWs of this study, HRTs of the VFCWs were very low (Gallinas *et al.*, 2013), due to the coarse sand granulometry coupled with the low algae influent concentrations. This fact did not allow having better *E. coli* removal whichever the HL applied.

Removal of microbial indicators in both VFCWs and HFCWs is a function of the HRT. For HFCWs microbial inactivation tends to be asymptotic. Therefore, after a certain value (around 2 Ulog) (*Torrens et al.*, 2010) an increase of HRT would not result in a significant higher removals for HFCWs. This could explain that the *E. coli* was not affected by HL in the studied HFCWs.

3.5. Conclusions

The viability of different designs of SSFCWs to upgrade pond effluent quality has been examined in two complementary study sites.

Two pond effluents have been fully characterised from a biological, physicochemical and microbiological perspective. The algae genera have been characterised using optical microscopy (with *Scenedesmus* spp. *Chlorella* spp. and *Euglena* spp. as predominant genera in facultative pond effluent and *Chlorella* spp. and *Euglena* spp. in tertiary pond effluents) and quantified via Chl-a and particle count analyses using laser granulometry. The laser granulometry technique for counting particle size has served to characterise the effluent from the lagoon, revealing the size of the predominant algal genera.

Pond effluents present large quantities of organic matter, good biodegradability $(BOD_5/COD \text{ around } 0.5 \text{ in facultative ponds})$ and have also shown a great variability. The effluents presented a seasonal behavior that had a strong correlation with solar irradiance and temperature. These two parameters affected the algae genera of the pond and its concentration and associated parameters (SS, COD, BOD₅ and SS).

A correlation between ChI-a and SS, BOD₅ and COD was also found (100 µg/L ChIa≈30 mg COD, ≈12 mg BOD₅ and ≈10 mg SS in facultative pond effluents and 100µg/L ChI-a≈37 mg COD, and ≈12 mg SS in maturation pond effluents). COD, dCOD, BOD₅, SS and turbidity were higher in warmer periods having high solar irradiance. On the other hand, ammonia and bacterial and viral indicators showed the opposite behavior. In periods with low algae concentrations, temperature and solar irradiance, the concentration of ammonia and bacterial and viral indicators were higher in the pond effluent. The concentration of algae, and related parameters (SS, COD) were higher in the facultative pond (case study 1) due to the low HRT of the tertiary pond pilot system (study site 2) and the lower concentrations of nutrients and organic matter in the pond inlet.

The studies have proved the viability of VFCWs and HFCWs for upgrading effluent from facultative and maturation ponds. Different conclusions have been drawn from each of the two study sites:

- Subsurface flow constructed wetlands performance: study site 1

- The study has demonstrated the effectiveness of VFCWs in retaining algae (SS removal 59-78%, Chl-a removal 48-83%) completing organic matter degradation (COD removal 35-62%) and nitrifying the facultative pond effluent (N-NH₄⁺ removal 71-92%) even in winter periods and for HL up to 80 cm/day. Retention of phosphorus was low and in one year the removals diminished drastically from all beds (from 80% to 20% for planted filters). Removal of bacterial indicators ranged from 1-2 Ulog and viral indicators ranged from 0.5 to 1 Ulog. Although all filters performed efficiently during the two years of monitoring, differences in performance were evident based on the design and operation.
- Frequent monitoring of infiltration rates is a useful tool to study the filters' hydraulic performance. Filters present good infiltration with general values >1.10⁻⁴ m/s when there are no algal deposits on the surface. Infiltration rates decrease in vertical flow constructed progressively with every successive batch (feeding). Tracer tests allow determining the mean hydraulic retention times and the detention time distribution curves in the vertical flow constructed wetlands. The mean HRT varies from 2 to 12 hours depending on the filter's design and operation.

- The influence of design parameters as well as operational parameters on filter performance has been stated. The depth of the filters, type of sand, hydraulic load feeding regime (fractionation of the hydraulic load) and resting period duration all affected the hydraulic performance and purification efficiency of the filters. The presence of plants did not significantly affect the filter performance (in terms of both physicochemical and microbiological parameters) although it was important in terms of maintenance and temperature moderation. The deeper filters presented better performance for all physicochemical parameters, especially for the removal of organic matter. Filter depth was also found to have a significant effect on algae. From the IRs and the tracer tests, no consistent conclusions could be made with regards to the hydraulic behavior based on the type of sand (crushed river sand). Regarding filter performance, crushed sand filters performed worse for physicochemical parameters than river sand filters in all the tested conditions.
- Generally speaking, the increase in hydraulic load reduced removal efficiency in all VFCWs: a considerable decrease in COD, SS and TKN removals was observed when doubling the HL. Hydraulic retention time was strongly influenced by the fractionation of the daily HL and also played an important role in determining the treatment level in the filters. During the first days of feeding, COD, SS and TKN performances were significantly improved at higher fractionation values, in all of the 65-cm filters.
- The removal of microbial indicators in VFCWs depended mainly on the water retention time in the filter, which in turn depended on the depth of the filter, the hydraulic load and the dose volume per batch. The presence of plants did not significantly affect the removal of indicator microorganisms, indicating that the presence of *Phragmites* is of minor importance for the removal of microorganisms in intermittently dosed VFCWs. No significant differences were found between the two types of sand tested (crushed and river sand). Low temperatures did not limit the removal of indicator microorganisms in VFCWs. Bacterial indicators were removed at a higher rate than viral ones. Somatic coliphages were removed at higher rates than F-specific bacteriophages.
- Failure to respect the recommended feeding and resting periods (3-4/7 days) coupled with the presence of algae resulted in clogged filters in Case study 1. Clogging by algae strongly decreased IR since almost all of the algae and SS were filtered at the surface if the particles were larger than 20-30 µm. Ponded water at

the surface hindered oxygenation, an anoxic state resulted and performance in terms of nitrogen and dCOD decreased. On the other hand, SS removal and algae retention increased. Ponded water favored more algae development and increased clogging. Therefore, control of the feeding and resting period is of great importance for the durability and the reliability of VFCWs.

The sand granulometry of d10≈0.25, CU≈4.7 was adequate for the studied conditions. The best performance was found for the river sand filters planted with *Phragmites* 65-cm depth. To avoid clogging, feeding/resting periods of 3-4/7 days are appropriate. This requires three beds in alternation, and having maximum hydraulic and organic loads on the operating filter of 80 cm/day and 170 gCOD/m²·day, respectively. These dimensional bases may be useful for upgrading existing facultative ponds or for the design of new facultative ponds.

- Subsurface flow constructed wetlands performance: study site 2

- The study demonstrated the effectiveness of VFCWs and HFCWS to improve tertiary pond effluent quality by retaining algae (SS removal 29-58%, Chl-a removal 34-59 for VFCWs; SS removal 58-71%, Chl-a removal 61-69% for HFCWs) and completing organic matter degradation (COD removal ranging from 42-54% for VFCWs and 45-47% for HFCWs). The VFCWs filters nitrified the pond effluent (N-NH₄⁺ removal 53-65%) and TN removal was low (around 20%) due to the formation of nitrates. HFCWs removed total nitrogen (43-62%) and ammonia (35-59%). Moreover, both VFCWs proved the capacity to remove bacterial indicators (*E. coli* removal averages of 0.8-0.9 Ulog for VFCWs and 1.3-1.7 Ulog for HFCWs).
- Although removal of algae and SS in VFCWs were no high on average, the percentage of SS removal was high (between 67-73%) when SS concentrations were above 40 mg/L SS in the filters inlet. For low algae inlet concentrations (SS < 40 mg/L) HFCWs were more effective than VFCWs.
- VFCWs with sand granulometry of d₁₀≈0.5 and CU≈1.9, did not provide the anticipated performances for VFCWs of 70 and 100-cm depth. No clogging episode was detected, with feeding/resting periods of 3.5/3.5 days, and with maximum hydraulic and organic loads on the filter in operation of 75 cm/day and 50 gCOD/m²·day respectively. However this tested sand particle size, coupled with the low SS content (due to low algae content), did not result in improved performance

for either the physicochemical or microbiological parameters. HFCWs did not experience any clogging with maximum hydraulic and organic loads of 40 cm/day and 30 gCOD/m²·day respectively.

 For VFCWs, the deeper filters presented better removals for all parameters. HFCWs with finer gravel (8/12 mm) presented significantly higher reductions for all parameters. The HL significantly affected the removal efficiency on SS, particulated COD and ammonia (a decrease in % removal when increased HL for both VFCWS and HFCWs). HL did not have a significant effect on the removal of *E. coli*, and dCOD.

4. SUBSURFACE FLOW CONSTRUCTED WETLANDS FOR SWINE SLURRY TREATMENT

4. SUBSURFACE FLOW CONSTRUCTED WETLANDS FOR SWINE SLURRY TREATMENT

4.1. Introduction

4.1.1. Problem statement

Land spreading of nitrogen-rich swine slurry poses a significant threat to surface waters trough non-point source pollution. Agro-alimentary activities produce high-strength wastewaters with marked seasonal fluctuations in terms of quantity and quality. Untreated swine slurry contains considerable amounts of non-stabilized organic matter and high concentrations of ammonium that can reach values of 8000 mg/L, depending on the farm characteristics (Obaja *et al.*, 2003). Spreading excess slurry over croplands may result in contamination of groundwater and eutrophication of surface waters. Once distributed on the fields, ammonium nitrogen, the main form of nitrogen in slurry, is readily oxidized into nitrate, which is poorly absorbed by soil colloids. Swine slurry nutrients in excess of crop uptake can accumulate and even saturate soils. At saturation, nutrients are lost to either surface or ground waters (Martinez *et al.*, 2009).

In many areas of Europe, disposal of swine slurry is a serious problem for farmers. Reducing total nitrogen load is especially important in sensitive areas where aquifers are contaminated with nitrates due to the high volume of swine slurry applied to the lands, as is the case in several Mediterranean regions. In many cases, the harmful environmental effects of pig farm effluents are caused by its high concentration in a limited area and the defective management of these wastes.

The Code of Good Agricultural Practice aims at reducing and preventing water pollution by nitrates from agricultural activities to protect human health and aquatic ecosystems. Through the "Nitrate Directive" (91/676/EEC), the EU seeks to reduce water pollution caused or generated by nitrates from agricultural sources. EU Directive 91/676/ECC transposed into Spanish legislation through RD 261/96 of February 16 on the "protection of waters against pollution caused by nitrates from agricultural sources." The Directive imposes on the EC Member States the identification of vulnerable zones in those countries where action plans must be adopted to reduce nitrate leaching, either to surface water or groundwater. In these so-called "vulnerable areas," a maximum of 170 kg of N from animal wastes per hectare per year is allowed. Under Spanish Law 16/2002, the industries involved (farms) must obtain an Integrated Environmental Authorization, whose purpose is, among other environmental protection goals, to prevent problems derived from swine slurry management.

In addition to nitrogen leaching, another problem of swine slurry management is the low content of dry matter, usually in the range of 2-5%, which increases transportation cost, makes the application of these manures on land and crops difficult and limits the periods of application (Flotats *et al.*, 1999). Therefore, thorough treatment of swine slurry before discharge into bodies of water or for water reuse, or even partial treatment prior to land application, may be necessary in some situations (Harrington and Scholz, 2010).

Large swine slurry volumes are currently generated throughout the world, with estimates for Spain reaching approximately 5600 million tons (Vázquez *et al.*, 2013). Spain is the second country in the European Union in terms of pork production, with a total of over 26 million heads, followed by Germany. Catalonia's pig density reaches more than 7 heads per hectare, with a total of almost 7 million heads, equivalent to an annual production of over 1000 tons of meat. This is 5-6 times the production of the rest of the Spanish state. In vulnerable areas, applying discharge limits, one hectare of land can accommodate a maximum of about 50 heads' slurry. In areas of intensive production, the number of heads sometimes exceeds 1000 per hectare of available land. In such circumstances, there is a surplus of manure whose application to agriculture is not possible without pre-treatment (Teira, 2008).

Figure 4.1 shows both the geographical distribution of cattle manure in Catalonia and the nitrogen surplus generated.



Figure 4.1. Map of cattle manure and nitrogen surplus in Catalonia (modified from ACA, 2013)

The pig industry in many regions of the Mediterranean, including Catalonia, have hundreds of small producers whose limited production and low annual budgets do not allow for implementation of advanced technological waste treatment. This high number of small or medium size piggeries has created the need for a simple, suitable waste disposal technology capable of reducing the nitrogen load applied to land. Small industries, as well as small towns, encounter problems when choosing treatment plant methods that have similar fixed costs regardless of population size and the industry they serve. The high concentration of N in slurry makes treatment costs too high and sometimes limits the sector's development.

4.1.2. Swine slurry characteristics

Pig slurry is the mixture of pig manure, unused fodder because of the type of feeding and livestock management, spilled drinking water and water from cleaning and cooling operations. Sometimes, it also includes rainwater or surface runoff from the farm area. Its water content is high, so swine slurry has a liquid consistency (Casassas, 2004).

The composition of swine slurry varies greatly. The amount of nitrogen excreted per year on a farm depends on the number of animals, the type of farm and the animal species as well as from the diet of animals. The basic slurry components are shown in Table 4.1.

Parameter	Min	Max	Average
Total Solids (g/kg)	13.68	169	62.16
Volatile Solids (g/kg)	6.45	121.34	42.33
Percentage (VS/TS)	46	76	65
COD (g/kg)	8.15	191.23	73.02
N-TKN (g/kg)	2.03	10.24	5.98
N-NH4 ⁺ (g/kg)	1.65	7.99	4.54
Norg (g/kg)	0.4	3.67	1.54
N-NH4 ⁺ /N-NTK	57	93	75
Phosphates (g/kg)	0.09	6.57	1.38
Potassium (g/kg)	1.61	7.82	4.83
Copper (mg/kg)	9	192	40
Zinc (mg/kg)	7	131	66

Table 4.1. Pig slurry composition in Catalonia (modified from Campos et al., 2004)

One of the main features of slurry is the low carbon-nitrogen ratio, indicating low aerobic fermentation capacity. Slurry degrades slowly under anaerobic conditions and is considered an organic fertilizer of little organic value though high mineral content. The amount of organic matter can be around 65%. This amount of organic matter makes the material suitable for biogas generation and composting. Most of the nitrogen is present as ammonium, and as seen in the Table 4.1, nitrogen value is close to 75%. It has a basic pH of around 8, due to carbonates, ammonium and volatile fatty acids. Its conductivity is high, indicating the presence of ionized salts. Water content exceeds 90% and is a key factor to be taken into account when considering the cost of transport. It also contains secondary nutrients, micronutrients and heavy metals. Given its origin, fecal pathogens are present as well. The high metal content can be detrimental for its use in agriculture and to its own degradation because the high concentration of metals can be toxic for some bacteria, although this can be addressed simply by varying the animals' diet. The high phosphorus content in manure makes it a highly polluting agent for water sources (Bayona, 2013).

4.1.3. Swine slurry management and treatment

As mentioned above, the Nitrates Directive limits the amount of nitrogen that can be applied to land. This puts pressure on farmers in the EU to manage their wastewaters effectively to prevent eutrophication of surface water. The current volume of manure usually exceeds crop fertilizer needs. The directive promotes "appropriate N spreading calendars, sufficient manure storage for availability only when the crop needs nutrients and good spreading practices" (European Union, 1991). Building alternative storage and treatment units or additions to traditional treatment and operational methods is required to abide by the Nitrates Directive (Henkens and van Keulen, 2001). Despite restrictions to land spreading, it is likely to continue to be the most cost-effective method of pig manure disposal. Reducing the concentration and volume of nutrients from pig farm wastes is often essential. Swine slurry treatment is intended largely to solve this overproduction of nutrients in order to adjust the amount and quality to farmers' demand for organic matter and fertilizer.

Swine slurry management usually consists of three main phases:

- <u>Phase 1</u>: Solid-liquid separation, the primary treatment process used to improve liquid manure handling properties and to generate solids. Several methods are available to separate solids from liquids, including sedimentation (solids settle by gravity), mechanical separation (screen, centrifuge, screw press and belt press separators), evaporation ponds, dehydration, coagulation and flocculation (Borin *et al.*, 2013).
- Phase 2: Converting the solid fraction into an exportable product used for composting or generating biogas (methane) (Borin *et al.*, 2013). Composting consists of aerobic biological decomposition and stabilization of organic substrates under conditions that create thermophilic temperatures (between 50 and 70 °C). The required ratio of C/N for these systems is 25-35. Pig slurry has a ratio of around 9, so that extra carbon input for possible treatment is usually required. The high temperatures reached contribute to sanitizing manure by eliminating pathogens, weed seeds, insect eggs and larvae. Odors are also removed by decomposing volatile compounds, and weight, volume and humidity are reduced. In ideal conditions, there is no loss of total nitrogen, and part of the ammonium nitrogen is transformed into organic nitrogen.
- <u>Phase 3:</u> Reducing nutrient contents in the liquid fraction to meet discharge criteria or spreading the remaining nutrients on arable land. Nutrient contents reduction in the liquid fraction can be achieved by different techniques: anaerobic codigestion, activated sludge treatment, batch sequencing reactors,

ultrafiltration, reverse osmosis or natural technologies such as CWs, which will be explained in the next section.

4.1.4. Swine slurry treatment with constructed wetlands

The lack of solutions for sustainable and cost-effective treatment of agroindustrial wastewater is a widespread problem. Many farmers and farming industries lack large-scale treatment facilities to deal with excessive amounts of wastewater. Therefore, there is a need for low energy consumption technologies (including natural technologies: ponds, CWs, IP), easy for the farmer to manage and maintain, and having low construction, operation and maintenance costs.

CWs have been in use for about 30 years as a sustainable option for treating domestic and urban wastewater. Although they are mainly used for this purpose, the viability of these systems for removal of other type of wastewaters is being studied. As reported by Szogi *et al.* (1995), CWs have the potential to eliminate organic compounds and nutrients from piggery wastewater, as they efficiently remove suspended solids, biodegradable organic matter and pathogenic microorganisms. Nitrogen removal depends on the system's design, process configuration and loading rates (Kadlec *et al.*, 2000). Detoxification is another important issue, as CWs can remove heavy metals, persistent organic substances and emergent pollutants (Reyes-Contreras *et al.*, 2011). Using subsurface flow wetlands is becoming more common in the treatment of different types of wastewater from both industrial and agricultural sources.

Swine slurry is high-strength wastewater, so treatment with CW has usually required the implementation of pre-treatment operations or even influent dilution (Hunt and Poach, 2001). Treatment efficiency varies for different pollutants and changes considerably in space and time, depending mostly on the type of CW used, its design, age of the system, feeding mode (how wastewater is applied), HL and hydraulic HRT. Under the effect of different temperatures, treatment efficiency tends to change during the year (Politeo, 2013). Politeo (2013) has compiled experiences using CWs for pig farm effluents and created a "piggery wastewater treatment wetlands database" (PWDB). Existing treatment wetlands for swine slurry have a wide variety of configurations, designs, flow rates, and inlet qualities. The Table 4.2, prepared from the PWDB, summarizes the case studies.

Location	Wetland type	COD (r	ng/L)	TKN ((mg/L)	N-NH₄ ^⁴	(mg/L)	N-NO ₃	(mg/L)	TP (r	ng/L)	Reference
		IN	OUT	IN	OUT	IN	OUT	IN	OUT	IN	OUT	
Australia	SSFCW	629	399	261	183					19.4	15.7	Finlayson <i>et al</i> ., (1987)
USA	FWSCW			70	6	55	3.5			25.8	6.2	Hammer e <i>t al.,</i> (1993)
USA	FWSCW					24.1	7.15	2.18	2.88			Hunt e <i>t al</i> ., (1993)
USA	FWSCW			130	43.1	112	37.3			29	17.4	Cathcart <i>et al.,</i> (1994)
USA	FWSCW			54.4	16.2	45.1	13.1			25.8	13	McCaskey <i>et al</i> ., (1994)
USA	FWSCW			416	249	405	244	1.06	2.35	17.4	14	Reaves <i>et al.,</i> (1994)
China	SSFCW	1847	246									Wang <i>et al</i> ., (1994)
USA	SSFCW	667	421		201	155						Parkes <i>et al.,</i> (1998)
China	SSFCW	1865	246									Junsan <i>et al.,</i> (2000)
USA	SSFCW	808	464	175	109					73	55	Poach <i>et al.,</i> (2003)

Table 4.2. Summary of key data for 18 case studies (PWDB) (Politeo, 2013)

Median inlet and outlet concentration of COD, TKN, N-NH4⁺, N-NO3⁻ and TP for each case study

Location	Wetland type	COD (mg/L)	TKN ((mg/L)	N-NH₄	⁺ (mg/L)	N-NO ₃	(mg/L)	TP (I	ng/L)	Reference
		IN	OUT	IN	OUT	IN	OUT	IN	OUT	IN	OUT	
Lithuania	SSECW	374	68	31	19 1	16.6	11.6			9.6	0.8	Struseviĉius and
Lindania		0/1	00	01	10.1	10.0	11.0			0.0	0.0	Struseviĉiene (2003)
USA	FWSCW	808	464	175	109					73	55	Poach <i>et al.</i> , (2004)
USA	FWSCW			86	53					56	48	Stone <i>et al.</i> , (2004)
USA	FWSCW	308	148	63.8	19.6	40.8	12.4			51.8	49.4	Hunt <i>et al.,</i> (2007)
USA	FWSCW	445	246							71	66	Poach <i>et al.</i> , (2007)
Korea	Hybrid SSFCW	7100	6333	1492	1383	1408	1307			81	60	Kato <i>et al.,</i> (2010)
Spain	SSFCW	11656	9752	2629	1191	2028	1546	39	28	30.5	28.4	Sánchez-García <i>et al.,</i> (2010)

 Table 4.2. (continued) Summary of key data for 18 case studies (PWDB) (Politeo, 2013)

Median inlet and outlet concentration of COD, TKN, N-NH4⁺, N-NO3⁻ and TP for each case study

The majority of these case studies use FWSCW to avoid clogging and require a significant land area with an average system size of 0.14 ha. SSFW systems for piggery wastewater are primarily used in China and Europe. Within SSFCWs type, HFCWs has been most commonly used for swine slurry treatment. SSFCWs, mainly HFCWS, are susceptible to clogging, since the accumulation of solids shortens the effective life of a constructed wetland, making solids removal a necessary pre-treatment step. Upstream storage ponds or solid separators can remove solids and ideally release only liquid effluent for treatment in the wetlands. The author of the database (Politeo, 2013) studied a hybrid system to treat the liquid fraction of swine slurry after aerobic treatment with good results. Kato *et al.*, (2010) is the only case that studied the performance of a hybrid system in treating almost-raw piggery wastewater. In this study, the high TN concentration in swine effluents caused problems with respect to oxygen supply in the plant's vertical filters.

Most of the CWs experiments on the Table 4.2 treat pre-treated swine slurry, as can be seen with the organic matter and nitrogen concentrations (<100 mg/L COD and <100 mg/L TKN). These CWs are supplementary wastewater treatments, in combination with pre-treatments including ponds, vermifiltration, anaerobic digesters or even activated sludge reactors meant to reduce nitrogen by nitrification and denitrification (Meers *et al.,* 2008). Finally, some studies treat swine slurry by recirculation or adding runoff or tap water for diluing the ammonium content than can be toxic for plants (Harrington and Scholtz, 2010).

4.2. Specific objectives

The purpose of this study is to evaluate the viability of hybrid SSFCW to treat swine slurry to obtain a quality suitable for different land application or discharge into water bodies. The specific objectives to achieve this main goal are:

- To fully characterise swine slurry (partially settled with an upstream storage pond).
- To specifically design a hybrid pilot plant consisting of a VFCW and a HFCW to treat swine slurry and operate it for 20 months.
- To evaluate treatment efficiency of the hybrid SSFCW, monitoring nitrogen dynamics, physicochemical parameters, and bacterial indicators.
- To study the capacity of nitrification/denitrification of a hybrid system treating partially settled swine slurry.

 To study the influence on the VFCW's treatment efficiency and hydraulic performance of design parameters (presence of *Phragmites*), operational parameters (dosing and feeding modes) and deposits on filter surface.

4.3. Material and methods

4.3.1. Study site description

The study was carried out at a private pig farm, Can Corominas, in Viver i Serrateix, in the Berguedà region of Catalonia (Spain), at an altitude of 606.4 meters above sea level, with a Mediterranean climate and an average annual temperature of 12.9 °C and average daily temperatures ranging from 1 °C to 29 °C. Annual rainfall is 660 mm. The farm has an area of one hectare, and it accommodates approximately 580 sows and 2000 piglets up to 18 kg in closed bays.

The slurry is stored in pits down the ground, and once they fill up, it is emptied into two storage ponds with depths between 3 and 5 meters. Filling usually happens every 15 days, and the ponds are emptied twice a year by towed cisterns transporting slurry as organic fertilizer to adjacent field crops.

4.3.2. Pilot plant description

The pilot plant for this project was designed, built and operated based on modular systems (Plantdepur) designed by Spanish company MOIX (MOIX, Obres i Serveis, SL) together with the University of Barcelona (http://www.plantdepur.com/). The construction was carried out by the company using the modular beds designed initially for treatment of domestic wastewater. This modular system allows for transport and easy installation without much civil work, as well as easily extending the system's capacity. A diagram of a modular tank design used in some pilot plants is shown in Figure 4.2.



Figure 4.2. Modular tank design (HDPE tank)

The pilot plant is a hybrid CW made up of a vertical and a horizontal flow connected in series. The general design diagram of the pilot wastewater treating plant is shown in Figure 4.3.



Figure 4.3. General design of the pilot plant treating swine slurry

The VFCW modular tank was modified after 4 months from the initial design in HDPE to galvanized steel due to problems with lateral pressure (Figure 4.4).



Figure 4.4. First VFCW tank with problems of lateral pressure and the new tank

Pilot plant receives slurry from an accumulation pond with anaerobic nature. The slurry is pumped into the VFCW, whose waters are directed towards the HFCW. Finally, the treated water from the HFCW is diverted into a big slurry accumulation pond. The main design parameters of the hybrid pilot plant are shown in Table 4.3.

	HYBRID SYSTEM					
	VFCW	HFCW				
Pretreatment	Reservoir	VFCW				
Modular tank	Galvanize steel (3)	Galvanized steel (1)				
Tank size	4.6x0.7x0.6 m	4.6x2.3x0.6 m				
Filling media	30 cm gravel (2-8 mm) 20 cm gravel (20-30 mm) 10 cm gravel (20-60 mm)	Crushed brick (2-8 cm)				
Vegetation	2 beds with Phragmites australis 1 bed unplanted	Phragmites australis				

Table 4.3. Main design par	ameters for the hybrid CW
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The vertical system was designed based on the model developed by the IRSTEA in France (with several beds in parallel and operated intermittently and sequentially), and it can treat raw wastewater without pretreatment as long as rest periods and intermittent feeding are applied to prevent clogging (Molle *et al.*, 2006). The tank is divided into three equal, independent, isolated units with an area of 3.2 m² each. The filling media is the same for the three units. All filters have a with a total depth of 60 cm, consisting of a filtering layer of approximately 30 cm of 2-8 mm gravel, followed by a transition layer of 20 cm of 20-30 mm gravel and a drainage layer 10 cm deep, with 20-

60 mm gravel. One of the units is not planted with *Phragmites australis* to check the effect of macrophytes.

Swine slurry is supplied by means of a pump regulated by a timer. An overflow pipe was installed to control HL. The swine slurry is directed to a distributor where water is sent to any of the three distribution pipes that feed each VFCW unit. A tile is placed on the bed down the pipe outlet to prevent local erosion and help distribute water evenly across the surface. Water flows vertically and is collected at the bottom. The water from the VFCW is directed to the horizontal module by gravity through a pipe.



Figure 4.5. VFCW pilot: general view, distribution pipe and Phragmites australis

In the second HFCW module (4.7 x 2.3 x 0.6 m), water, which sequentially enters as it receives the effluent from the vertical filter, flows in a 14.1 m circuit. (Figure 4.6.). The tank is filled with crushed brick between 20 and 80 mm in diameter and planted with *Phragmites australis.* The outlet consists of a flexible pipe that controls the water column level in the wetland bed. Finally, the effluent exits through a pipe to a big swine slurry storage pond.



Figure 4.6. HFCW pilot: general view and Phragmites australis

4.3.3. Experimental protocol

The pilot plant was monitored and operated for 20 months (2011-2013). The operating conditions and monitoring are described in the next sections.

4.3.3.1. Operation

A summary of the operation parameters is shown in Table 4.4. The VFCW pilot received a HL of 5.6 cm/day (180 L/day). For the same 5.6 cm/day HL, two dosing regimes were applied varying the feeding schedule in order to study the effect of fractionation on filter performance. Thus, two operating periods were established: the first one of 1batch per day (180 L/batch), and the second one of 5 batches per day (36 L/batch) batches per day. Filters were fed for 7 days and left resting for 2 weeks. The way water entered the HFCW depended on the HRT of the VFCW units.

	Table 4.4. Operation parameters of the hybrid GW						
		VFCW	HFCW				
HL and	Period 1	HL 5.6 cm/day filter in operation 1 batch/day (1 batch: 5 cm/day)	HL 5.6 cm/day (from VFCW				
dosing	osing HL 5.6 cm/day filter in operation Period 2 5 batches/day (1 batch 1.9 cm/day)		outlet)				
Fee	ding	Pump (discontinuous)	Gravity (continuous or intermittent flow depending on the VFCW outlet flow)				
Oper	ation	Each unit is fed for 1 week and rest for 2 weeks	Continuous (no resting periods)				

Table 4.4. Operation parameters of the hybrid CW

4.3.3.2. Monitoring

Water quality monitoring

<u>Physicochemical analysis.</u> Physicochemical parameters were evaluated in each pilot plant component (VFCWS inlet, VFCWS outlet, HFCW inlet and HFCW outlet). Analysis of weekly grab samples was performed for 20 months. EC, pH, COD, dCOD, TKN, N-NH₄⁺, N-NO₃⁻, and P-PO₄³⁻ were analysed according to the Standard Methods (APHA, AWWA-WPCF, 2005). Methods of analysis references are listed in Apendix 1.

<u>Microbiological analysis.</u> Microbiological parameters were evaluated in each pilot plant component (VFCWS inlet, VFCWS outlet, HFCW inlet and HFCW outlet). Analysis of weekly grab samples was performed for 20 months. The bacteriological indicator (*E. coli*) was evaluated according to the Standard Methods protocol (APHA, AWWA-WPCF, 2005). Methods of analysis references are listed in Apendix 1.

<u>Sludge deposit monitoring</u>

Samples of sludge accumulated on the surface of the VFCWs were collected every 3 months to monitor the degree of mineralization and composition as fertilizer. DM, OM%, N% and P% were analysed according to MAPA, 1994. Methods of analysis references are listed in Apendix 1.

Hydraulic monitoring

In the VFCWs units, Infiltration Rates (IR) per filter were quantified by measuring the level of the surface water with an ultrasound probe as described in section 3.3.2.3.2. All data were recorded on a data logger minute by minute for 12 months. Every week, the recorded data were downloaded for evaluation.

Temperature monitoring inside the filters

Two temperature sensors placed at a depth of 15 cm were also installed in a planted VFCW unit and in the unplanted VFCW unit. The temperature sensors would contribute to explain the differences caused by the presence of vegetation. These data were recorded on a data logger minute by minute for 12 months. Every week, the recorded data were downloaded for evaluation.

Biomass inside the filter

Ammonium and nitrite oxidizing bacteria inside the VFCW units were identified by using the Nitri-Vit (Vermicon) test kit. Samples were collected from the filter at 15 cm depth after one week of operation and analyzed with this Nitri-Vit test using fluorescent microscopy.

4.3.3.3. Statistical analysis

Statistical analysis of the raw data was done using the statistics computer packages Excel 2010 and SPSS 16.0. for Windows. Excel 2010 was used for descriptive statistics (i.e., averages, SD) and to perform regression analysis. SPSS 16.0 was used to analyze variance (ANOVA). The level of significance (p) was established at p≤0.05. Data from these test parameters that were not normally distributed were log-transformed to present the normal distribution required to run these analyses.

4.4. Results and discussion

4.4.1. Influent characterisation

The average, maximum and minimum concentrations of VFCWs influent pollutants are shown in Table 4.5. Influent wastewater presented the typical characteristics of swine slurry (*Lee et al.*, 2014), with high concentration of SS, organic matter, nitrogen and phosphorous. However, most CW studies, as shown in Table 4.5, present lower influent pollutant concentrations (most CWs operate with pre-treated wastewater). Only Sánchez-García (2010) and Kato *et al.* (2010) apply a similar high-strength influent.

Parameter	VECW influent quality		
	Average	Min	Мах
EC (mS/cm)	15.2	13.4	19.3
рН	7.6	7.4	8.1
SS (mg/L)	4123	765	19537
DM (g/L)	12.1	7.1	21.2
BOD₅ (mg/L)	2124	520	4750
COD (mg/L)	6985	3514	16785
dCOD (mg/L)	3125	875	12874
TKN (mg/L)	2345	1038	3752
N-NH4 ⁺ (mg/L)	1876	1078	2654
N-NO3 ⁻ (mg/L)	31	0	102
P-PO4 ³⁻ (mg/L)	267	62	874
<i>E. coli</i> Ulog (CFU/100ml)	6.3	5.4	8.4

Table 4.5. VFCWs influent quality

The influent presents high variability. Pig manure storage in the basin can be considered the pre-treatment step, as it has anaerobic conditions. During the study, different levels of solids accumulation were observed (depening on the settling).

Therefore, even though the pump was placed in order to pump the liquid fraction, solid fractions were sometimes also applied to the VFCWs.

The average SS content was 4000 mg/L with high variations (from approximately 1500 to 10000 mg/L) as a result of each bed (filter in operation) receiving an average of 230.8 g of SS/m²·day, and 76.9 g of SS/m²·day for the whole VFCW pilot, which represents approximately 28 kg of SS/m²·year. This value is similar to SDRBs applied with primary or secondary sludge (Troesch, 2009). French VFCWs treating raw wastewater receive much lower loads (about 150 g of SS/m²·day with the filter in operation. Most of the CWs experiences treating swine slurry also applies lower SS surface loading rates than this study.

The average value of influent DM was 12 g/L. Similar concentrations of DM are found in the literature for different types of sludge applied to SDRBs: Koottatep *et al.* (2005) applied sludge from emptying septic tank (with a DM content of 12 g/L) to SDRBs in Thailand, and Burgoon *et al.*, (1997) describes in his study with SDRBs the application of sludge from an aerated pond with a DM content of 14 g/L. The DM value of 12 g/L represents 87 kg of DM/m² per year (filter on operation) and 27 kg of DM/m² per year (entire VFCW). These values are similar of those applied in SDRBs. Nielsen et *al.* (2005) recommend a maximum of 60 kg of DM/m² per year for the all SDRBs. SDRBs are implemented to treat sludge from activated sludge systems, primary sludge and septage from emptying septic tanks (Molle, 2014).

Organic loads presented a high variability in the study, ranging from 3600 to 14000 mg/L of total COD with a very high average organic load of 391.2 g of COD/m²·day (filter in operation) and 130.4 g of COD/m²·day (the whole system) depending, again, on the farm operation and storage conditions. About 50% of the COD was dissolved COD. The BOD₅/COD ratio presented values of 0.25-0.42. Ratios greater than 0.5 are considered to easily treatable by biological means. Ratios below 0.3 are difficult to degrade biologically (Tchobanoglous *et al.,* 2003). The influent entering the VFCWs presents intermediate biodegradability.

Regarding nitrogen forms, influent is characterized by high N-NH₄⁺ concentration (most of the nitrogen is ammonium, typical in raw swine slurry). As shown in Table 4.5, during the monitoring period, TKN influent concentration ranged from 1100 to 270 mg/L, with a median load of 131.3 g TKN/m²·day (filter in operation) which was higher than almost all the values reported in the literature for CWs (Borin *et al.*, 2013). Only Sánchez-
García (2010) and Kato *et al.* (2010) presents similar values. N-NH₄⁺ influent concentration ranged from 1000 to 2100 mg/L, which represents a load of 105.1 g N-NH₄⁺/m²·day, again, higher than almost all the values reported in the literature. The high concentration of ammonium caused problems for plant development, so the filters performed almost like unplanted filters. Sánchez-García (2010) also report problems in macrophyte growing in their studies.

It seems that the ammonium content was toxic for the *Phragmites*, which barely developed. Hunt *et al.* (2004) indicate that high organic loading rates can increase the risk of ammonium toxicity in some constructed wetland plants. The most commonly used macrophytes for all type of wastewaters and in the studies of CWs treating swine slurry are *Phragmites* and *Typha* (Scholz, 2006). More recently, *Glyceria* has being used because of its high tolerance to ammonium toxicity (Tylova-Munzarova *et al.*, 2005). Wastewater dilution with clean water, or recirculation to improve nutrient removal, is a common practice in constructed wetland operation, particularly if the wastewater can be slightly toxic for plants. Heavily polluted wastewater can also be diluted by less contaminated waters such as roof runoff (Scholz, 2006).

Phosphate variability was very high and may be attributable to storage conditions and farm management. The concentration of *E. coli* was high, around 6.3 ULog, slightly higher than in urban wastewater.

4.4.2. Performance of the hybrid pilot

4.4.2.1. Effluent quality and removal of pollutants

Table 4.6 presents the average, maximum and minimum influent and effluent concentration (mg/L) and removal efficiency (%) in the hybrid CW for the physico-chemical parameters.

Denemeter		Inlet			VFC	W *			HFCW			TOTAL REMOVAL
Parameter –	Average	Min	Max	Average	Min	Max	%	Average	Min	Max	%	%
EC (mS/cm)	15.2	13.4	19.3	13.4	3.4	18.7	-	12.4	8.1	15.1	-	-
рН	7.6	7.4	8.1	8	7.3	8.3	-	8.1	7.5	8.7	-	-
SS (mg/L)	4123	765	19537	956	165	4023	76.8	235	16	671	75.4	94.3
DM (g/L)	12.1	7.1	21.2	6.6	6.4	7.5	45.5	5.2	5.1	5.8	21.2	57.0
BOD₅ (mg/L)	2124	520	4750	945	324	2465	55.5	520	19	1041	45.0	75.5
COD (mg/L)	6985	3514	16785	3245	1865	10245	53.5	1820	444	3985	43.9	73.9
dCOD (mg/L)	3125	875	12874	2465	345	6214	21.1	1130	133	2633	54.2	63.8
TKN (mg/L)	2345	1038	3752	1345	647	2374	42.6	715	120	1485	46.8	69.5
N-NH₄⁺ (mg/L)	1876	1078	2654	1132	598	1812	39.7	698	172	1058	38.3	62.8
N-NO₃⁻(mg/L)	31	0	102	439	41	564	**	154	12	425	64.9	**
P-PO4 ³⁻ (mg/L)	267	62	874	117	15	284	56.2	49	6	120	58.1	81.6

Table 4.6. Hybrid CW performance: average, maximum and minimum influent and effluent concentration and % removal of pollutants

*Average for the three VFCWs, ** Nitrification

The VFCWs presented good removal percentages, in general, for all the parameters. The SS concentration in the filters'outlet was much lower, confirming the great filtering capacity of VFCW, with removal rates of about 75%. HFCW after the vertical filters greatly upgraded the quality of SS, thus reaching a final quality of approximately 230 mg/L. The long HRT of the HFCW allowed sedimentation of almost all SS.

Organic matter removal (BOD_5 and COD) was about 50% in the VFCW, despite the high influent load. Oxidation of organic matter was very high, despite the big organic loads. The HFCW after the VFCW also had good removal rates (about 50% on average), thus reaching a final removal rate for the whole system of 74% for dCOD and 64% for COD.

The vertical flow filters also nitrified the piggery wastewater, with TKN and N-NH₄⁺ removal of about 40% and producing high concentration of nitrates, thus confirming the filters oxidation capacity thanks to batch feeding and resting periods. The HFCWs reduced total nitrogen, as the system partially denitrified the HFCW inlet nitrates (VFCW inlet). The average removal rates for the HFCW were 46.8% for TKN, 38.3% for N-NH₄⁺ and 64.9% for N-NO₃⁻. The final effluent contains an average 598 mg/L of N-NH₄⁺, 715 mg/L of TKN and 154 mg/L of N-NO₃⁻, despite the high inlet concentration of swine slurry. Section 4.4.2.2 (nitrogen compounds profile) details the behavior of nitrogen compounds in the hybrid pilot.

Phosphorus was reduced averaging 86% removal. It is noteworthy that after 10 months of operation, removal decreased to 30% due to saturation of phosphorus adsorption, so it is expected that such reduction will decrease more in the next few years. A longer monitoring period is needed to study the efficiency of phosphorus removal in these systems. Phosphorus removal is closely related to the physical, chemical and hydrological properties of the filtering material, as it is physically or chemically adsorbed mainly by ligand exchange. However, a decrease in the concentration of soluble inorganic phosphorus is linked to biological activity, either by macrophyte assimilation or removal through microbiological processes (Molle, 2003). In both processes, adsorption and biological phosphorus uptake, accumulation capacity is finite. As the wetland reaches its stable state, when it is no longer capable of adsorbing if plants are not removed any more phosphorus, elimination decreases dramatically (Vymazal, 2007).

Removal of phosphorus by the plants (plant uptake) in the VFCWs can be considered negligible as the plants developed poorly. The effect of the plants on treatment performance will be described in section 4.4.2.4. Part of the phosphorus was also retained and accumulated in the sludge on the top of the filters, as will be explained in section 4.4.2.6.

Table 4.7 shows the average *E. coli* concentration and removal throughout the study.

		VF	CW*	HFC	w	
	Inlet (LogCFU/100mL)	Outlet (LogCFU/100mL)	Removal Ulog	Outlet (LogCFU/100mL)	Removal Ulog	TOTAL REMOVAL Ulog
E. coli	6.3	5.3	1	3.6	1.7	2.7

Table 4.7. E. coli concentration (Log CFU/100mL) and removal (Ulog)

*Average for the three VFCWs

Regarding microbiological indicators, *E. coli* was reduced on average 1 Ulog in the VFCW. Similar reductions were found in the other studies with VFCWs of 65-100 cm depth (Chapter 3). However, filters of 30 cm with fine sand (Chapter 3) (Torrens *et al.*, 2009a) presented lower removal rates than these VFCWs. Those shallow filters always showed removal rates <1 Ulog. The higher removals observed in the case of the VFCWs could be explained because of sludge accumulation on the top of the filters. This sludge layer diminishes infiltration rate (as will be explained in section 4.4.2.3), thus increasing retention time. Hydraulic retention time is a key factor in removal of bacterial indicators. The higher the retention time, the higher the removal (Torrens *et al.*, 2010). Moreover, this sludge layer can increase filtration capacity, thus enhancing *E. coli* removal by filtration.

The HFCW removed 1.7 Ulog *E. coli*, which is within the usual range of HFCW removal (between 1-2 Ulog) (Huertas, 2009; Sasa, 2014). The combination of HFCW and VFCW reached 2.7 Ulog in total, providing an outlet quality of about $3x10^3$ CFU/100 mL of *E. coli*.

Effluent quality of the hybrid system was not suitable for discharge in bodies of water, but important elimination of nitrogenous components, as well as of organic matter, phosphorus, and even some disinfection, is achieved. The hybrid system has a dual function: it is a process of solid-liquid separation and biological treatment at the same

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time. In the VFCW, part of the pollutants is retained in the deposit layer on the surface of the filters. The characteristics and mineralization of the solid fraction are detailed in section 4.4.2.6.

4.4.2.2. Nitrogen compounds profiles

In terms of nitrogen, the influent is basically composed of ammonium and a small part of organic nitrogen. Around 80% of the nitrogen present in the slurry is in the form of ammonium. Figure 4.7 shows the average hybrid CW influent and effluent nitrogen forms.



Figure 4.7. Average nitrogen forms in the hybrid system

In the VFCWs effluent, the content of ammonium and organic nitrogen has decreased considerably. Part of the influent nitrogen is retained in the sludge accumulated on the filter surface (section 4.4.2.6.). Plant uptake could also be considered negligible as the plants did not properly develop. Moreover, the results shown in section 4.4.2.4 (Effect of plants) do not show any differences in nitrogen removal rates. The main nitrogen removal in these VFCWs is achieved by biological processes that convert organic and ammonium nitrogen into nitrate in an aerobic environment (nitrification). To be really efficient in nitrification, the filters should be in fully aerobic condition, which means that the sand must be kept quite continuosly unsaturated and drained. To distribute the effluent evenly over the whole filter surface, a batch feeding system must be installed together with a distribution network. This ensures that the effluent to be treated will

reach all filter points as fast as possible to avoid both over and under loaded areas. There is proof that batch feeding enhances aeration by convection, which acts as a plug flow to push the gas below the water level and aspirate the air from the atmosphere over it, once the filter surface is free of water. The batch operation provides aeration of the gravel substrate and exposes the internal biofilms to atmospheric oxygen. During the cycle's drain phase, atmospheric oxygen causes enhanced ammonium and organic matter oxidation (Kadlec and Wallace, 2008).

The Nitri-vit kits analysis showed the presence of ammonium oxidizer (Figure 4.8) and nitrite oxidizers microrganisms (Figure 4.9) inside the VFCW filters, demonstrating the filters' oxidation capacity despite the high organic loads applied.



Figure 4.8. Fluorescence image (ammonium oxidizers)



Figure 4.9. Florescence image (nitrite oxidizers)

Nitrifying bacteria are sensitive to environmental changes. Among the inhibiting factors are high concentrations of ammonium, low temperatures, a pH out of the range between 6.5 and 8.6 and an amount of dissolved oxygen below 1 mg/L (Ogden and Campbell, 1999). It is well known that temperature plays a critical role in metabolism rate of the bacterial species and is a limiting factor in the transformation processes of nitrogen compounds. Nitrification was affected by temperature, as shown in Figure 4.10. In this figure, dependence on nitrification temperature is observed, as registered by the submerged probes.



Figure 4.10. Correlation of $N-NH_4^+$ removal with temperature inside the filters (HL=5.6 cm/day, 1 batch/ day)

The correlation index shows dependence in the removal of nitrogen. The correlation of nitrification and temperature results in higher percentages of elimination with increasing temperature.

Figure 4.11 shows TKN removal (g TKN/m²·day removed) of the VFCWs filter according to two temperature ranges (<10 °C and >10 °C). The slope for the >10 °C range is higher than for the <10 °C, confirming that TKN removal is much higher at temperatures inside the filters >10 °C.



Figure 4.11. Effect of temperature on TKN removal (HL=5.6 cm/day, 1 batch/ day)

In the area of the study, winter temperatures are very low, reaching easily 0 °C in winter time. Therefore, these systems can operate much more efficiently in warmer areas, where a filter with similar characteristics and operation would remove much more TKN/m²·day. If such high TKN removal is not required, the total filter area could be smaller.

HFCWs remove the total nitrogen by nitrifying and denitrifying reactions. Removing nitrogen, and especially ammonium nitrogen, is a two-step mechanism. This is a microbiological process for closing the biogeochemical cycle of nitrogen and release it into the atmosphere. Ammonium is oxidized by autotrophic bacteria into nitrate in the presence of oxygen. The nitrate is then reduced in the denitrification step under anoxic conditions, to molecular nitrogen by the action of heterotrophic bacteria (Cooper, 2005; Platzer, 1999). Almost all organic nitrogen entering in the HFCW is removed (92 %) and a 38% of the entering ammonium. More than 50 % of nitrates are denitrified in the HFCW (Figure 4.12).



Figure 4.12. Nitrate load per m² in the influent and effluent HFCW (HL=5.6 cm/day, 1 batch/day)

Denitrification is a sequential process in which nitrates are gradually transformed into nitric oxide, nitrous oxide, and finally, molecular nitrogen that is liberated into the atmosphere. Heterotrophic microorganisms responsible for this process are mainly aerobic facultative, able to adapt to the environmental conditions in which they find themselves. Heterotrophic microorganisms need a source of organic carbon to oxidize. A proper ratio of C/N and readily biodegradable organic matter are essential for good kinetics of the reaction. These nitrification-denitrification processes are possible in the HFCW thanks to the presence of enough organic matter (and that carbon source) in the VFCW effluent. The optimum pH for denitrification is between 7.0 and 8.5 (the average pH in the inlet HFCW was 8, with a minimum of 7.3 and maximum of 8.3).

Therefore, the pH conditions were favorable for the denitrification process in the HFCW.

Average total removal of TKN and N-NH₄⁺ by the hybrid CW was 69.5% and 62.8%, respectively. Similar yields were found by Borin *et al.* (2013) in a hybrid system with notable TKN and N-NH₄⁺ reductions of 64% and 63%. This is significant, but in our case study, nitrogen and organic loads/m².day were much higher.

Total nitrogen reduction (TKN + N-NO₃⁻) was, on average, 63.4 %. Therefore, the results show that nitrogen removal by the hybrid CW has effectively reduced the nitrogen content of the pig slurry, thus reducing existing production surplus in this farm. The percentage of this pollutant reduction reached in the study would allow a higher volume of effluent application to agricultural land and enabled more sustainable management of swine slurry in the farm.

Considering a 63.4% reduction of total nitrogen, the hybrid CW surface required per pig head was roughly estimated. Assuming an average volume of swine slurry of 2 m³/year with 3 kg/m³ of nitrogen, the required hybrid CW surface would be approximately 0.11 m² per head. As indicated above, the percentage of nitrogen removal is strongly related to temperature, so the value obtained can be reduced with the increase of average temperature.

Apart from land application, there is another scenario to consider that is to obtain a quality of effluent that could be able to discharge into bodies of water. This could be achieved in two ways:

- Addition of a second HFCW module in series. With the C and N concentration of the HFCW effluent, another module of HFCW could be incorporated in series to reduce the total nitrogen and achieve limits close to those of discharge into bodies of water.
- Influent recirculation. Another option is to dilute the influent by recirculation to reduce the ammonium content that can be toxic to the plants, like the Harrignnton and Scholtz (2010) experience in Ireland.

4.4.2.3. Vertical flow constructed wetland hydraulics

The VFCW pilot received an average HL of 5.6 cm/day (180 L/day for a batch height of 5.6 cm). For most of the study (12 months), the filters received 5.6 cm/day in one application. For 4 months, the same HL was applied fractioned into five applications of 36 L each, for a batch height of 1.9 cm. Filters were fed during 7 days and left resting for 2 weeks throughout the study.

As explained in Chapter 3, continuous monitoring of IRs was a very useful and practical tool to study the filters' hydraulic behavior. The three VFCWs beds presented similar average IRs, ranging from 10^{-4} m/s to 10^{-7} m/s depending on the day of feeding, operation and temperature. Similar values (though slightly higher) ranging from 10^{-4} m/s to 10^{-6} m/s were found SDRBs treating primary non-digested sludge (Dabo, 2004).

During one week of feeding, the IRs decreases (Figure 4.14). The same phenomenon was observed in SDRBs treating primary non digested sludge (Torrens *et al.*, 2006a) or with the IRs studies of VFCWs treating pond effluent explained in Chapter 3. Fast infiltration at the beginning of each feeding period can be related to differences due to the low humidity conditions inside the filter beds after resting. While filters are fed and become humid, the infiltration rates decrease.

In the studied operation conditions, no clogging problems were detected thanks to fixed feeding/resting periods established. With the studied organic loads, the filters' filtration capacity was good for 7 days in winter periods. However, more than 7 days of feeding reduced infiltration capacity to values in the range of 10^{-7} m/s, as shown in Figure 4.13. Hence, the feeding/resting cycle of 1 week/2 weeks seems to be well adapted for the studied conditions, with 7 being the maximum number of feeding days for VFCWs with air temperature between 1 and 15 °C for the applied organic loads. In warmer periods (with air temperatures between 15 °C and 30 °C), the infiltration capacity during the seven feeding days remained good (about 10^{-5} m/s) (Figure 4.13). The high temperatures allow better drying and mineralization of the organic deposits, so that water can infiltrate better. In summer, infiltration capacity was not reduced as much in the 7 feeding days, so in areas with higher temperatures, feeding periods could be slightly longer with no risk of clogging.



Figure 4.13. Infiltration rates during one week in the VFCWs (1 batch/day, December and May)

4.4.2.4. Effect of the presence of *Phragmites*

As reported above, *Phragmites australis* hardly developed at all, so no differences were found between the planted and unplanted beds (in removal efficiency and hydraulics). Table 4.8 shows the effluent water quality and removal results for the three beds (two planted and one unplanted).

	Outlet quality				Removal %			
-	Average 3 beds	V1-P	V2-P	V3-NP	Average 3 beds	V1-P	V2-P	V3-NP
EC (mS/cm)	13.4	13.2	13.6	13.4	-	-	-	-
рН	8	7.9	8	8	-	-		
SS (mg/L)	956	934	987	946	76.8	77.3	76.1	77.1
DM (g/L)	6.6	12	12.1	12.1	45.5	0.8	0.0	0.0
BOD ₅ (mg/L)	945	921	980	935	55.5	56.6	53.9	56.0
COD (mg/L)	3245	3245	3204	3287	53.5	53.5	54.1	52.9
dCOD (mg/L)	2465	2510	2416	2471	21.1	19.7	22.7	20.9
TKN (mg/L)	1345	1328	1307	1401	42.6	43.4	44.3	40.3
$N-NH_4^+$ (mg/L)	1132	1072	1149	1174	39.7	42.9	38.8	37.4
N-NO3 ⁻ (mg/L)	439	441	420	457	*	*	*	*
P-PO4 ³⁻ (mg/L)	117	108	98	145	56.2	59.6	63.3	45.7

Table 4.8. Performance of planted and unplanted filters: average outlet pollutant concentration and removal %

V1-P (filter 1 planted), V2-P (filter 2 planted), V3-NP (filter 3 unplanted), * nitrification

4.4.2.5. Effects of the dosing and feeding modes

Figure 4.14 shows the effects of feeding mode on infiltration rates (1 batch/day or 5 batches/day). For the same hydraulic load of 5.6 cm/day, when fractionation is higher (5 batches/day), infiltration rates are lower. When fractionation is higher, the water pressure is lower due the lower batch volume, and moreover, humidity is higher inside the filter because the time elapsed between applications is shorter. All this results in lower infiltration rates as the water percolates slowly.



Figure 4.14. Effects of fractionation of the HL on infiltration rates

Only COD removal was significantly affected (p<0.05) by the fractionation of the HL (Figure 4.15).



Figure 4.15. Effects of fractionation of the HL on COD removal efficiency, (HL=5.6 cm/day), (temperatures between 5-10 °C)

COD was better at higher load fractionation. These results match the results of Torrens *et al.* (2009a). COD removal and nitrogen oxidation appeared to be highly dependent on fractionation. For the same HL, with higher fractionation by decreasing the batch

volume, retention time is increased, and there is greater exchange of the less mobile fraction of the pore water. This circumstance affords closer and longer contact between media and pollutants and results in higher purification, as seen in Chapter 3. Nevertheless, the difference with the VFCWs experience described in Chapter 3 is that nitrification was not affected by fractionation of the HL. Although fractionation can increase retention time in the filters, it can diminish oxygenation, so the removal of nitrogen compounds is not affected.

4.4.2.6. Characterisation and effects of deposits on the surface of the vertical flow constructed wetlands

Part of the dry matter, organic matter and nutrients contained in the applied slurry is retained on the vertical filter surface. The sludge on top of the vertical filters was dried and mineralized in the bed, as happens in the first stage of the French VFCWs treating raw wastewater or SDRBs. This biosolids organic layer appears to be a key component that can favor treatment performance or limit some processes. Indeed, this layer can improve filtration efficiency and, thus, solids removal, water retention time into the system and treatment performance, as long as the media stay in aerobic conditions, and it can reduce permeability of the filter and improve water distribution on the filter's surface. It also allows water to flow in the entire filter volume at lower velocity, favoring ammonium adsorption nitrified between batches or during resting periods, and it is the place of major biological activity, once its thickness becomes significant (Molle, 2014). Nevertheless, hydraulic and organic loads, as well as operating conditions (batch feeding, alternation between feeding and resting periods), have to be closely controlled to encourage deposit mineralization. Otherwise, the deposit layer can generate process limitations, such as waterlogging, oxygen transfer limitation (convection and diffusion) and decreasing biosolids mineralization. All these processes are interdependent (Molle, 2014).

Dry matter content of the accumulated sludge varied from 40% to 60% depending on the season (40% of DM in winter and 60% in summer). However, the quality of the sludge was stable, with contents in the range of 40% VS, 3% TKN, and 1 % P_2O_5 (Table 4.9).

	%DM	%VS	%TKN	%P ₂ O ₅	%N-NH₄⁺/%N-TKN
Average	45.7	40.5	3.2	1.1	65
SD	17.3	4.1	0.8	0.3	12.2

 Table 4.9. Sludge composition (average and SD) throughout the period (samples taken one week after resting)

Percentages of VS, TKN and P_2O_5 were constant during the study. DM content varied, mainly depending on the temperature and the loads applied. Percentage of DM contents in other studies is reported as: 30% DM and 40-50% VS, with SDRBs treating sludge from activated sludge systems (Uggeti, 2011). Nutrient values of 2-3% of TKN and around 1% of TP are found in the sludge accumulated on the surface of SDRBs treating primary sludge from a settling tank (Dabo, 2004).

This residue accumulates (and dries and mineralizes) over time, generating a volume that directly affects the system's hydraulics, as explained in section 4.4.2.3., so it is an important aspect to take into account in the evolution of treatment system design and management for possible final application of this sludge. Dry sludge, which is about 1% of the total mass of the fresh slurry, could be used for compost or applied to the land as fertilizer with additional analysis.

At the end of the study, about 25 cm of sludge were accumulated on the filters. Figure 4-17 shows sludge accumulation on the surface (cm) of V1-p and V3-np filters for 7 months.



Figure 4.16. Accumulation of sludge on the surface of the filters measured by ultrasound probes

The values registered by the installed sensors, in addition to providing information about the hydraulic system performance (IRs), have allowed us to estimate the amount of sludge accumulated on the surface of the vertical filter, and the slope of the regression line is identifiable as the ratio of deposit accumulation on the surface (approximately 19.4 cm/year). This value matches the observed values using a ruler placed on the filters' wall (around 1-2 cm/month). The deposit layer created in the first stage of the French VFCW accumulates at an average rate of 2.5 cm/year at nominal load. Kootttatep *et al.* (2005) reports accumulations of 12 cm/year for SDRBs treating septage from septic tank emptying. Troesch (2009) displays a compilation of experiences with SDRBs treating different sludges or wastes and reports accumulations per year ranging from 15 cm to 60 cm/year, depending on design, load, operation and type of sludge or influent.

4.5. Conclusions

The viability of using a hybrid SSFCW to treat swine slurry has been studied.

Influent wastewater presented the typical characteristics of swine slurry, with high concentration of SS, organic matter, nitrogen and phosphorous. The SS average content was 4000 mg/L, as result the VFCW received 76 SS/m²·day, representing approximately 28 kg SS/m²·year. These values are similar to those of the SDRBs treating sludge.

During the monitoring period, TKN influent concentration ranged from 1100 to 270 mg/L, with a median load of 131 g TKN/m².day (filter in operation), higher than almost all values reported in the literature for CW experiences. The influent is composed mainly of ammonium and a lower content of organic nitrogen. Around 80% of the nitrogen present in the hybrid system influent appears in the form of ammonium. N-NH₄⁺ influent concentration ranged from 1000 to 2100 mg/L, representing a load of 105 g NH₄⁺/m²·day. Ammonium content was toxic for *Phragmites*, which barely developed in the VFCWs.

Overall, the VFCWs had good removal rates for all of the parameters. SS concentration in the filter outlets was much lower, confirming the great filtration capability of the VFCW, with removal rates of approximately 75%. The HFCW used after the vertical filters greatly upgraded the quality of the SS, having a final quality of approximately 230 mg/L.

Organic matter removal (BOD₅ and COD) was approximately 50% in the VFCW. Oxidation of organic matter was very high, despite the higher organic loads. The HFCW after the VFCW also had a good removal percentage (about 50% on average), thus attaining a final removal percentage for the overall system of 74% for dissolved COD and 64% for total COD. Phosphates were reduced with an average removal of 86% (56% for VFCW and 62% for HFCW) and after ten months in operation, removal decreased to 30% due to saturation of phosphorus adsorption.

Regarding the microbiological indicators, *E. coli* was reduced by an average of 1 Ulog in the VFCW. The HFCW removed 1.7 Ulog. The combination of HFCW and VFCW achieved 2.7 Ulog in total, thus providing an outlet quality of approximately 3x10³ CFU/100 mL of *E. coli*.

The VFCWs also nitrified the swine slurry, removing approximately 40% of TKN and N-NH₄⁺ and producing high nitrate concentrations, thus confirming the filters' oxidation capability thanks to the batch feeding and resting periods. The HFCWs reduced total nitrogen, as the system partially denitrified the nitrates of the HFCW inlet (VFCW outlet). The average removal rates for the HFCW were 46.8% for TKN, 38.3% for N-NH₄⁺ and 64.9 % for NO₃⁻. The final effluent contains an average of 598 mg/L of N-NH₄⁺, 715 mg/L of TKN and 154 mg/L of N-NO₃⁻, despite the high inlet concentrations of the swine slurry. The correlation between nitrification and temperature resulted in a higher percentage of elimination when temperatures increased.

On average, nitrogen reduction (TKN + NO_3) was 63.4 % in the overall hybrid system. The results reveal that nitrogen removal by the hybrid CW effectively reduced swine slurry nitrogen content to levels that are under the existing production surplus in intensive farms. The percentage of reduction of pollutants reached in the study allowed for a higher effluent volume in agricultural land and more sustainable swine slurry management at the farm.

Considering the average of 63.4% removal of total nitrogen (with a volume of swine slurry of 2 m^3 /year and 3 kg/m³ of nitrogen), the required surface of hybrid CW would be approximately 0.11 m^2 per animal. This area may be reduced in areas with higher temperatures.

Effluent quality of the hybrid system was not suitable for discharge in bodies of water, but a significant elimination of nitrogenous components, as well as of organic matter, phosphorus, and even some disinfection, was achieved. The hybrid system had a dual function: it simultaneously carries out solid-liquid separation and biological treatment. In the VFCW, some of the pollutants were retained and mineralized in the surface deposit layer.

The VFCW pilot received an average HL of 5.6 cm/day with no clogging problems, with feeding and resting periods of one and two weeks, respectively. With the studied pollutants loads, and under these operating conditions, the filter infiltration capacity remained good for up to 7 days in the winter. After seven days of feeding, infiltration capacity reduced to values in the range of 10⁻⁷ m/s. Thus, seven is the maximum number of feeding days for VFCWs with air temperatures between 1 and 15 °C. Fractionation of the HL decreased infiltration rates in VFCWs, but it only significantly affected COD removal.

The deposit accumulated on the surface of the VFCW was dried and mineralized in the bed. Dry matter content of the accumulated sludge varied from 40% to 60%, depending on the season (40% DM in winter and 60% in summer). However, the quality of the sludge was stable, with contents of approximately 40% VS, 3% TKN, and 1% P_2O_5 . Accumulation of deposits on the surface was found to be approximately 20 cm/year. This biosolid organic layer improved filtration efficiency, and thus solids removal, water retention time in the system and treatment performance.

The use of a hybrid system (VFCW + HFCW) allowed for a reduction of the overall nitrogen load for the swine slurry, thanks to the combined nitrification/denitrification processes. VFCWs operated intermittently (batches) and with sequential feeding (1 week feeding/2 weeks resting) resulted in good hydraulic performance without clogging problems, despite high pollutant loads. The modularity of the pilots allows for easy installation of this technology.

5. SUBSURFACE FLOW CONSTRUCTED WETLANDS FOR CAR WASH EFFLUENT TREATMENT

5. SUBSURFACE FLOW CONSTRUCTED WETLANDS FOR CAR WASH TREATMENT

5.1. Introduction

5.1.1. Problem statement

Europe has a long history in water management, and more specifically in "small water cycles"; the management of drinking water supply, sewerage and treatment of wastewater are well-developed practices. However, the recycling of treated wastewater has not been widely applied in most European countries. Due to the increasing need to protect water resources, the growth in environmental awareness and the public's inclination to promote sustainability, the pressure to use reclaimed water is gradually increasing (de Koning *et al.*, 2008).

In some Mediterranean regions such as Catalonia, urban development is putting their existing water resources at risk. In these zones, any attempt to reclaim, recycle, and reuse water is considered a "win-win" strategy by both enhancing water supplies and reducing pollution. Such win-win strategies can be implemented in many industrial sites and for activities that involve tap water consumption (Al-Odwani *et al.,* 2007).

One sector that contributes to high water consumption is commercial car washing. Currently, vehicle washing facilities are widely spread throughout all urban areas in developed countries. Despite their significant environmental impacts that result from the high consumption of resources (potable water and electricity) and generation of waste, there are very few facilities that are committed to using innovative solutions to address this problem. Car wash stations are one of the industrial applications that consume large quantities of fresh water on a daily basis and could benefit from recycling programmes. Improved water use efficiency is, in its simplest form, a reduction in water needs.

In an environmental context, this efficiency concept must be extended to include considerations of water quality. Efforts to improving water use efficiency should be placed in conjunction with maintaining or improving water quality. Therefore, consumption and pollution should be reduced by better management and technical

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improvements in the treatment and recycling of wastewater. The car wash industry poses additional environmental threats through its use of detergents.

The implementation of car wash water reclamation, recycling, and reuse promotes the preservation of limited water resources in conjunction with water conservation and watershed protecting programmes. Despite their potential, water reclamation, reuse, and recycling technologies are greatly underused (Al-Odwani *et al.*, 2007). Professional car wash water reclamation has been in use and has grown to be more advanced over the last decade. Water reclamation is getting more attention from regulators and manufacturers as a means of conserving water and controlling water quality. The management techniques and technologies used to treat and reclaim car wash water are shown in Section 5.1.3.

Regarding legislation, the car wash industry appears today to be more conscious of the need for wastewater treatment and water reclamation. Environmental legislation and guidelines concerning this specific issue have been released worldwide. Examples show that in Queensland, Australia, it is mandatory to use, at most, 70 L of fresh water in a single car wash, and some European countries restrict water consumption to 60–70 L per car and/or impose reclamation percentages (70–80%). In the Netherlands and Scandinavian countries, 60–70 L/car is the maximum amount of fresh water consumption allowed. The recycling of 80% of car wash effluent is compulsory in Germany and Austria (Zaneti *et al.*, 2011).

The majority of car wash facilities in Europe and Spain do not recycle their wastewater; they treat the wastewater in order to meet the established thresholds to connect to the sewage or to discharge to the receiving media. Currently, car wash facilities in the city of Girona use tap water, and each car wash consumes approximately 320 L of water (Lequia, 2008).

The criteria for vehicle wash reclamation systems must include public acceptance, aesthetic quality, microbiological risk and chemical issues. Reports by the International Carwash Association indicate that the water quality of vehicle washes must be sufficiently high such that the vehicles and wash equipment are not damaged (chemical risks include corrosion, scaling and spot formation), the microbial risk to operators and users must be minimal, and the aesthetic conditions must be acceptable (Zaneti *et al.*, 2012). Therefore, controlling the microbiological risk of reclaimed water is an important issue in the car wash industry. In addition to bacterial indicators, Legionella is an

important parameter that must be controlled if the installation has any equipment that can produce aerosols.

5.1.2. Car wash effluent characteristics

Car wash process constitutes the following steps: (1) application of degreasing agent all over the surface of the automobile (2) addition of acid and alkaline cleansers and (3) a coating (Páxeus, 1996). The main pollutants in wastewater from the car wash industry are described in Figure 5.1.



Figure 5.1. The main pollutants in wastewater from the car wash industry (Janik and Kupiec, 2007)

Effluents from car wash facilities contain a number of pollutants such as sand, dust, detergents/surfactants, organic matter, oil, fat, oil/water emulsions, carbon, asphalt and salts (Hamada and Miyazaki, 2004). This effluent also presents high levels of turbidity, organic matter, phosphorous compounds, nitrogen compounds, plasticizers, brake dust from rubber linings and various heavy metals (Janik and Kupiec, 2007; Zaneti *et al.,* 2012). Few studies have characterized car wash effluents, and even fewer from a microbiological point of view. However, the study conducted by Zaneti *et al.*, 2012

demonstrates that car wash effluent include high concentrations of bacterial indicators (fecal coliforms).

5.1.3. Management and treatment of car wash effluents

The main technical improvements carried out in car wash facilities in Europe have focused on providing top quality wash services with the best finish for maximum customer satisfaction. Companies that are pioneers in the sector have started working on optimizing and reducing the consumption of resources by seeking the following two objectives: 1) the economic aim of reducing production costs and adjusting prices to make their car wash systems more competitive, and 2) responding to the environmental concerns of the customers and joining the current trend of promoting sustainability and environmental responsibility. This aim places such companies in a position of preference within the market due to the value added by the provided services. To achieve this, companies have modernised and adapted their washing equipment, and optimised their operational parameters for more efficient water, energy and other supplies consumption. At the same time, the detergent and finishing product industries have also been upgrading their products to offer consumers with more environmentally friendly products and comply with the limitations established by current legislation.

Although biological treatment methods are widely used to treat urban sewage in municipal wastewater treatment plants, such technologies are usually not applied to the treatment of car wash effluent. This is due to drawbacks related to the characteristics of car wash wastewater: biodegradability, system's implementation (high investment and maintenance costs), or microorganisms' sensitivity to chemicals and temperature variations. Natural technologies have been never been applied to treat these effluents, even though CWs are used to treat effluents with similar characteristics (urban runoff) or those that are much less biodegradable (petroleum and oil industry) (see section 1.3).

Conventional treatment methods such as series of settler tanks and hydrocarbon separators are often used when wastewater needs to be discharged into the media or connected to sewage systems. If water reclamation is envisaged, the effluent must be treated to meet an acceptable level of water quality such that it can be recycled; thus, higher quality effluents are needed. In the car wash industry, a typical approach used for water reclamation systems entails physical-chemical treatment, i.e., flocculation-

sedimentation and direct filtration. Some providers have developed car wash recycling equipment based on floculation-coagulation processes and compact filtration systems. According to Brown (2002), car wash wastewater reclamation requires the separation of sand, gravel, oil and fat prior to reuse. Additional treatment processes can be employed to strengthen the quality of the reclaimed water such that it can be used in different wash stages (pre-soak, wash, rocker panel/undercarriage, first rinse, and final rinse). Some processes and technologies that have been proposed and tested include ultrafiltration. reverse osmosis. nanofiltration. flocculation-sedimentation and flocculation-flotation (Zaneti et al., 2011). Filtration treatment with activated carbon, ozone and ultrafiltration are being studied along with electrochemical methods such as anodic oxidation with diamond and lead dioxide anodes (Panizza and Cerisola, 2010; Kiran *et al.*, 2015).

Most reclamation systems that have been installed meet the needs of an individual operator reduce tap water consumption, control water and sewer hook-up costs, meet regulatory demands or some combination of these factors. The circumstances faced by operators and their desire to reduce tap water consumptiom or/and reduce the pollutants load to the sewage will dictate the choice of methods and installation of reclamation equipment. Some factors that the operators should take into consideration include: the nature of the contamination that must be treated, the concentration of pollutants, the volume of water used per day, the flow rate per minute used for different processes within the car wash station and the chemicals and procedures used in the wash or rinse process (Al-Odwani *et al.*, 2011). According to Partzsch (2009), decentralized water reuse schemes are considered to be "more green" or "eco-friendly", as they allow water to be treated and processed in a more nature-oriented way.

5.2. Specific objectives

The purpose of this study is to evaluate the viability of different SSFCWs configurations to treat the effluent from car wash facilities for internal recycle and reduce tap water consumption. The specific objectives to achieve this main goal are:

- To fully characterise the car wash effluent quality.
- To specifically design two SSFCW pilots (one VFCW, one HFCW) to treat the car washing facility effluent and operate it for 12 months.

- To specifically design one IP pilot to treat the car wash facility effluent and operate it for 12 months to compare with the SSFCWs pilots performances.
- To evaluate treatment efficiency of the three technologies, monitoring common (physico-chemical parameters, bacterial indicators and Legionella) and specific pollutants (hydrocarbons, fats and oils and detergents).
- To study the influence of operational parameters (hydraulic load, dosing and feeding regime) on the treatment efficiency and hydraulic behavior of the pilots.

5.3. Material and methods

5.3.1. Study site description

The Ramon Noguera Group Foundation has a special work centre with car wash facility. "Rentat de Vehicles de Montfullà", is located in the Montfullà Industrial Park in Bescanó (Girona, Spain). The car wash opened in June 2011, and it can work with any type of vehicle (private and commercial). The area's average yearly rainfall is between 700 and 900 mm, and average monthly temperatures range from 1° to 24° C. The Montfullà car wash station has currently operating two washing modules: car wash tunnel and a vehicle gantry washer.

- <u>Car wash tunnel</u>: The tunnel is an automatic car wash system in which the car is automatically carried through the tunnel with brush rollers and pressure water nozzles. The car wash tunnel includes:
 - 1. <u>Pre-wash</u>: a descaling product is applied with a hand-held lance and left to work of a few seconds 3L/vehicle; next, high pressure water is applied with a hand-held lance 20L/vehicle.
 - 2. Washing:
 - Shampoo arch and active foam arch (shampoo solution application -15-20 L/vehicle).
 - Brush arches (water with shampoo -150/200 L/vehicle). Part of the water used in the brush arches can be recycled and part is tap water. According to the supplier, 75% is recycled water and 25% is potable. According to project studies, during the first year of

operation the ratio was 50-50% approximately. However, nowadays very often due to problems in the recycling system, more than 75% of water used is tap water.

- i. Rinse arch (fresh water rinse 20 L/vehicle).
- 3. Finishing
 - Shining/protective wax arch (Wax solution application 30 L/vehicle)
 - Reverse osmosis water arch (final rinse with reverse osmosis water -30L/vehicle).
- 4. Drying section

Total water consumption is approximately 300L/vehicle (the average consumption per vehicle measured in 2014 was 278 L). All this water used in the process is tap water, except for the water in the brush arch that is sometimes partially recycled. Wastewater obtained from the system is collected and treated differently, depending on whether it is to be recycled or not. In the tunnel, water from pre-wash, shampoo arch, active foam, arch brushes and rinsing is collected and treated specifically to be recycled. The remaining water from the process, i.e., water from the finishing arches (shining, protective wax and reverse osmosis water) is collected and, after minimal required treatment, is discharged into the sewer system. Therefore, tunnel wash wastewater is collected in two different circuits:

- CIRCUIT A (recycling): consists of pretreatment with three settling tanks (ST) 10000L each, in series, followed by a recycling process by means of a reclamation module (sand filter). The part of the water that is not recycled goes into an oil-water separator, and it is finally discharged into the sewer system.
- CIRCUIT B (direct discharge): consists of pretreatment with a ST of 5000L followed by an oil-water separator to subsequently discharge the pretreated water to the public sewer system.
- <u>Truck and bus gantry washer</u>. The gantry washer is a structure consisting of brush rollers and pressure water nozzles. The water used in the gantry washer

is collected and treated. Part of the water from the truck and bus washing process is recycled together with the water from part of the tunnel wash process. The average commercial vehicle water consumption is around 400 L/vehicle.

5.3.2. Pilot plant description

The experimental plant located in the Montfullà car wash was built in January 2014. It includes three pilots (HFCW, VFCW and IP) (Figure 5.2). The three pilots treat the first ST wastewater. A pump installed in this first ST provides a maximum flow of 4.8 m³/h up to the first distribution or feeding point; i.e., the VFCW. From the pump, a main feeding pipe splits in 2 circuits: an auxiliary microbe feeding system or direct feeding system to the three pilots. The current study has been conducted with the direct feeding circuit. The main pipe allows water application to the pilot plants by branching out in three points:

- 1. VFCW inlet
- 2. IP system inlet
- 3. HFCW inlet

A solenoid valve was installed in each pilot.



Figure 5.2. Pilot plants layout

5.3.2.1. Vertical flow construced wetland pilot

The VFCW is made up of a container filled with filtering and draining material. The container is movable (Figure 5.2). The VFCW pilot plant treats wastewater directly from the ST. Wastewater is discontinuously applied on the surface though a distribution system (Figure 5.2.). The water percolates through the VFCW, and it is collected by a draining system connected to the sewer. Figure 5.3 shows a view of the VFCW pilot. Table 5.1 shows a summary of the general characteristics of the VFCW pilot.



Figure 5.2. VFCW distribution system



Figure 5.3. View of the VFCW pilot

	VFCW				
Operation	Discontinuous (by batches)				
Feeding mode	Instant flow at the application point 4.8 m ³ /h				
Distribution system	Overground pipeline with 6 outlets				
Container	Steel				
	The container has a total surface of 10.58 m ²				
	- Total pilot length: 4.6 m				
Container size	- Total pilot width: 2.3 m				
	- Height: 1.3 m				
	Calibrated sand 0.8 m high; 2 layers:				
	- 40 cm of calibrated sand (d_{10} =0.23, CU=3.2, fines content < 3%				
Filtering material	- Sand granulometry curves are shown in Appendix C				
	50 cm of 2-8mm gravel				
	Transition layer 10 cm (8-12mm gravel)				
Draining system	Draining layer 20cm (25-40 mm gravel				
Vegetation	Phragmites australis				

Table 5.1. Summary of VFCW pilot characteristics

5.3.2.2. Infiltration-percolation pilot

The IP line consists of a disk filter (DF) system, a storage tank with a pump system and a container filled with the filtering and draining materials (IP). The container is movable (Figure 5.4).



Figure 5.4. IP container and drip system

The DF system (Figure 5.5.) consists of four filters (120 mesh). The filters were installed in parallel. This system was designed for two filters to be working together while the other two are resting.



Figure 5.5. Disk filter system before the IP

Therefore, the wastewater from the collection point (ST) goes through two of the four ring filters towards the 1800 L storage tank. The tank has a submerged pump. This way, the wastewater to be treated in the IP pilot is applied under the surface through a drip irrigation system (subsurface drip irrigation). The water percolates through the IP and is collected by a draining system connected to the sewer system. Figure 5.6 shows a view of the IP pilot. Table 5.2 summarises the general characteristics of the system.



Figure 5.6. View of the IP pilot

	IP
Operation	Sequential (feeding and resting periods) and discontinuous (by batches)
Pre-treatment	4 disk filters (120 mesh) installed in parallel
	Submerged pump feeding.
Distribution system	From a pipe, 8 pipelines with 2.3L/h self-compensating drips are distributed.
/Feeding mode	Subsurface drip irrigation systems, at 10cm from the surface and separated 40
	cm between lines and 30 cm between emmiters.
Container built in	Steel
	The pilot has a total surface of 10.58 m ² ,
	- Total pilot length: 4.6 m
Tray Size	- Total pilot width: 2.3 m
	- Height: 1.3 m
	100 cm of sand (d ₁₀ =0.35 mm; CU=2.6, fine content <3%)
Filtering material	Sand granulometry curves are shown in Appendix C
	Transition layer 10cm (7-12 and 3-7 mm mixed gravel)
Draining system	Draining layer 20cm (25-40 mm gravel)
Vegetation	Grass: Zulueta Seed Compact mix (10% Lolium perenne, 5% Poa pratense and 85% Festuca arundinacea)

Table 5.2. Summary of pilot IP characteristics

5.3.2.3. Horizontal flow constructed wetland pilot

The HFCW pilot system consists of an elevated storage tank that receives wastewater from the ST by means of the main pump. This 1000L tank had to use gravity to send water into the HFCW. The feed flow was manually regulated by opening or closing a valve. However, it was not useful to adequately regulate the flow because solids clogged it. These solid deposits caused a progresssive decrease in flow and made it impossible to control and regulate it. In early July 2014, a peristaltic pump was installed to feed the HFCW and in order to better regulate the inflow. This container is divided into compartments (Figure 5.7). Water is applied under the surface in the inlet area. The water flows through the HFCW and is collected on the opposite side. The outlet system have and adjustable pipe to regulate the level inside the HFCW. From the HFCW, the treated water can be discharged into the sewer system by gravity or be pumped to the IP system. Figure 5.8 shows a view of the HFCW pilot. Table 5.3 shows a summary of the general features of the HFCW pilot.



Figure 5.7. HFCW container and outlet device



Figure 5.8. View of the HFCW pilot

	HFCW
Operation	Continuous
Feeding mode	Gravity and peristaltic pump
Container built in	4mm steel; interior compartments of 2 x 0.6m every 0.6m
Size	The pilot has a total surface of 10.58 m ² - Total pilot length: 4.6m - Total pilot width: 2.3m - Height: 0.6 m
Filtering material	Inlet and outlet areas (100 cm of 25-40 mm gravel respectively) Filtering zone (12-18mm gravel)
Vegetation	Phragmites australis
Outlet device	Adjustable level pipe

Table 5.3. Summary of pilot HFCW features

5.3.3. Experimental protocol

The experimental plant started operating in March 2014, and has been monitored for 12 months.

5.3.3.1. Operation

The three pilots have operated in parallel during the entire monitoring period. The pilot VFCW operated discontinuously (by batches). The high flow at the application points (4.8 m³/h) totally floods the filter surface. Wastewater to be treated is applied through a distribution system made up of aboveground pipes with six outlets. For the VFCW pilot, three HL were tested for periods: 1, 2 and 3. For each HL, two dosing modes were tested for a different number of applications (4 or 8 batches per day). Table 5.4 shows a summary of operating conditions of the VFCW pilot.

	Dosing modes (number of batches)	Influent flow (L/day)	HL (cm/day)
Period 1.1 2 months	4 (2 minutes/application)	640	6.2
Period 1.2 1.5 months	8 (1 minutes/application)	640	6.2
Period 2.1 1.5 months	4 (6 minutes/application)	1920	19
Period 2.2 2 months	8 (3 minutes/application)	1920	19
Period 3.1 2 months	4 (12 minutes/application)	3840	36
Period 3.2 2 months	8 (6 minutes/application)	3840	36

Table 5.4.	Operating	conditions	of the	VFCW	pilot

The IP pilot also operated discontinuously (by batches). Before the pilot, the pretreatment with the DF working in parallel operated to eliminate the larger solids so as not to clog the drip irrigation system. IP feeding was performed by means of a submerged pump placed in the 1800L deposit tank located after the disk filters. This pump is activated by means of a timer. From the feeding pipe, 8 underground selfcompensating drip lines with a 2.3L/h flow supply water to the device. The total flow they provide is 239.2 L/h. The IP pilot worked sequentially in feeding/resting cycles (5 feeding days/2 resting days). This resting period was applied to prevent sand clogging. 3 HLs were tested for periods 1, 2 and 3 respectively. As for the VFCW pilot, each HL was tested with a low number of applications (4) and a higher number of applications (8). Table 5.5 shows a summary of the operation conditions of the IP pilot.

	Dosing modes	Influent flow	HL
	(number of batches)	(L/day)	(cm/day)
Period 1.1 2 months	4 (30 minutes/application)	478	4.5
Period 1.2 1.5 months	8 (15 minutes/application)	478	4.5
Period 2.1 1.5 months	4 (120 minutesapplication)	1914	19
Period 2.2 2 months	8 (60 minutes/application)	1914	19
Period 3.1 2 months	4 (236 minute/application)	3764	36
Period 3.2 2 months	8 (118 minute/application)	3764	36

Table 5.5. IP pilot operation conditions

From 01/12/2014 to 22/12/2014, the IP was not 100% operational because the drippers were partially clogged. They were replaced with drippers of similar characteristics with a manual purge system to prevent them from clogging again (Figure 5.9).



Figure 5.9. Dripper purge cocks

The HFCW operated from 04/07/2014 to 04/07/2015 by gravity. In early July the peristaltic pump was installed to feed the HFCW and be able to regulate the inflow more effectively. For each pilot, three HLs were tested for periods 1, 2 and 3 respectively. Table 5.6 shows a summary of the operating conditions of the HFCW pilot.

		•	
	Average		
	Inflow	HL	
	(L/day)	(cm/day)	
Period 1	150 1	1 /	
3 months	150.1	1.4	
Period 2	762.9	7.5	
3 months	/02.0	7.5	
Period 3	4 400 0		
6 months	1493.3	-14	

 Table 5.6. Operating conditions of the HFCW pilot

5.3.3.2. Monitoring

Water quality monitoring

Water quality was monitored by systematically taking samples from the pilots (inlets, outlets and intermediate sampling points). The samply frequency was weekly, biweekly, monthly or bimonthly depending on the parameter and the sampling points: pilot's inlet, VFCW pilot outlet, IP inlet (sample taken from the IP's inlet tank), IP outlet, HFCW outlet, HFCW piezometer 1 (near the inlet) and HFCW piezometer 2 (near the outlet). The parameters were analysed by using a multiparametric probe: pH, EC, water temperature, redox, turbidity and dissolved oxygen (DO) or in laboratory (LABAQUA S.A.): COD, dCOD, DBO₅, SS, VSS, TKN, N-NH₄⁺, N-NO₃⁻, P-PO₄³⁻, sulphates (SO₄²⁻), chlorides (CI⁻), calcium (Ca²⁺), magnesium (Mg²⁺), alkalinity, total surfactants (anionic, cationic, non-ionic), oil, fats, hydrocarbons, *E. coli* and *Legionella* spp. Additionally one annual sample was taken at the pilots' inlets for helminth eggs (*Ancylostoma, Trichuris* and *Ascaris*). Table 5.7 shows a summary of sampling points and frequency of sampling.
	Sampling points						
Control parameters	Inlet	VFCW outlet	IP inlet	IP outlet	HFCW outlet	PIEZ.1	PIEZ.2
рН	W	W	W	W	W	М	М
EC	W	W	W	W	W	Μ	М
Temperature	W	W	W	W	W	Μ	М
Redox	W	W	W	W	W	Μ	М
Turbidity	W	W	W	W	W	Μ	М
DO	W	W	W	W	W	М	М
COD	W	W	W	W	W	М	М
dCOD	W	W	W	W	W	М	М
BOD₅	W	W	W	W	W	М	М
SS	W	W	W	W	W	М	М
E. coli	W	W	W	W	W	М	М
VSS	BW	BW	BW	BW	BW	-	-
TKN	BW	BW	BW	BW	BW	-	-
$N-NH_4^+$	BW	BW	BW	BW	BW	-	-
N-NO ₃ ⁻	BW	BW	BW	BW	BW	-	-
P-PO4 ³⁻	BW	BW	BW	BW	BW	-	-
Alkalinity	BW	BW	BW	BW	BW	-	-
S-SO42-	М	М	-	М	М	-	-
Cl	М	М	-	Μ	М	-	-
Ca ²⁺	М	М	-	Μ	М	-	-
Mg ²⁺	М	М	-	Μ	М	-	-
Anionic surfactants	BW	BW	BW	BW	BW	-	-
Cationic surfactants	BW	BW	BW	BW	BW	-	-
Non-ionic surfactants	BW	BW	BW	BW	BW	-	-
Hydrocarbons, oils and fats	BM	BM	-	BM	BM	-	-
Legionella spp.	BM	BM	-	BM	BM	-	-
Nematoda eggs	А	-	-	-	-	-	-

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able 3.7. Summa	arv or the sampling pro	locol (sampling points.	

W: weekly; BW: biweekly; M: monthly; BM: bimonthly; A: annual

The analytical methods are shown in Appendix A.

Hydraulic Monitoring

Hydraulic monitoring was carried out by means of pilot inlet and outlet flow controls. Inlet flow controls were done by calculating the time the pumps at each pilot's inlet were working. In addition, periodic manual controls (instant flow registration with a stopwatch and a test tube) were carried out to verify these values. HFCW inlet flow was checked systematically every week to be able to verify the proper functioning of the peristaltic feeding pump. Outlet flows were checked manually by registering instant flow with a stopwatch and a test tube. For each tested HL outlet flows were controlled for several hours. For VFCW and IP, outlet flow was controlled every 15 minutes: from a few minutes before a batch was applied until the next application. For HFCW these controls were carried out every 15 or 30 minutes for several hours.

5.3.3.3. Statistical analysis

Statistical analysis of the raw data was done using the statistics computer package Excel 2013 for descriptive statistics (i.e., averages, SD, % below detection limit).

5.4. Results and discussion

5.4.1. Influent characterisation

During the study period, the pump that feeds the pilot system was placed between 60 and 120 cm from the bottom of the ST. Therefore, the wastewater that was pumped was partially settled in relation to the direct effluent coming directly from the car wash facility. Table 5.8 shows pilot influent wastewater quality (average values, SD, maximum and minimum values, detection limit (dl) for each parameter technique and % of the number of samples below the ld (%<dl).

Parameters	Units	Average	Мах	Min	SD	ld	% <ld< th=""></ld<>
pH		8.0	8.9	6.7	0.4	0	0.0
Redox	mV	88.4	249.5	11.7	50.8	2000	0.0
EC	µS/cm	503.2	1259	179.0	143	0	0.0
DO	%	14.5	65.2	0.0	14.7	-	
DO	mg/L	1.2	5.6	0.0	1.3	0	41.0
Turbidity	FNU	85.0	265.0	33.8	45.8	0	0.0
COD	mg/L	48.8	158.0	bdl	35.0	10	2.0
dOOp	mg/L	22.3	100.0	bdl	20.0	10	12.0
PCOD	mg/L	30.8	98.0	5.0	19.7	-	-
BOD ₅	mg/L	14.0	50.0	bdl	10.3	5	4.0
SS	mg/L	41.0	138.0	bdl	33.5	3	0.0
VSS	mg/L	15.4	122.0	bdl	25.6	3	0.0
TKN	mg/L	4.2	34.2	bdl	7.8	3	12.0
N-NO ₃ ⁻	mg/L	2.5	14.8	bdl	3.4	0.5	6.0
N-NH4 ⁺	mg/L	0.3	3.0	bdl	0.7	0.1	16.0
P-PO4 ³⁻	mg/L	0.4	5.2	bdl	1.2	0.1	17.0
S-SO4 ²⁻	mg/L	51.4	157.3	31.8	40.0	5	0.0
Cl	mg/L	58.0	250.1	20.1	73.8	10	0.0
Ca ²⁺	mg/L	54.3	59.7	50.6	3.3	2	0.0
Mg ²⁺	mg/L	8.8	9.5	8.5	0.4	2	0.0
Alkalinity	mg/L CaCO₃	140.5	167.7	58.1	22.6	5	0.0
Anionic surfactants	mg/L	bdl	0.9	bdl	0.2	0.1	17.0
Cationic surfactants	mg/L	bdl	0.1	bdl	0.0	0.2	19.0
Non-ionic surfactants	mg/L	bdl	0.6	bdl	0.1	0.2	17.0
Hydrocarbons, oil and fats	mg/L	0.2	0.3	bdl	0.1	0.1	0.0
E. coli	CFU/100 mL	1262	20000	0	3669	-	-
Legionella spp.	CFU/L	0	0	0	0	-	-
Nematode eggs	Eggs/10L	0	0	0	0	-	-

 Table 5.8.
 Influent wastewater characterisation

Max=maximum, Min=minimum, SD=standard deviation, %<Id= percentage below Id

As table 5.8 shows (especially in average values and SD, as well as minimum and maximum values), the car wash effluent shows great differences throughout the year of the study. Theis great variation is also characteristic of raw urban wastewater. All parameters, especially SS, show significant variations, possibly due to variable dirt levels contributed by each vehicle and type of vehicle (cars, trucks), and, more importantly, to the time of sampling. If sampling was done immediately after a car is washed, the amount of settled material is lower. As explained above, the water entering the filters was partially settled. Four additional wastewater characterisations campaings were made in samples taken directly before the ST. These samples

presented about 10 % of VSS in reference to SS (SS values about 977 mg/L and VSS of 112 mg/L). Therefore, non-organic SS were about 90% (basically sand and fines). Settling of these particles was quick.

The BOD₅/COD ratio was close to 0.3, which is a medium biodegradability index, lower than urban wastewater (usually above 0.4) (Tchobanouglous et al., 2003). SSFCWs have never been applied to treat car wash effluents, however CWs are used to treat effluents with similar characteristics (urban runoff) or effluents that are much less biodegradable (from petroleum and oil industry) (Vymazal, 2014). The average percentage of VSS in reference to SS in the influent to the pilots was about 36%, which shows a significant amount of mineral solids (mineral solids adhered to car wheels and tires). These values are different from urban wastewater or other types of industrial wastewater (e.g., agroalimentary industry), which have a higher percentage of organic material and are more biodegradable. This type of wastewater, will, therefore, be more difficult to degrade biologically than urban or industrial wastewater with higher BOD₅ contents. In addition to biodegradability, other mechanisms such as filtration and sedimentation will be important to treat this water. This also means that this small amount of pollution that passes to the pilots will not be degraded by natural technologies. Long-term studies would be necessary to analyse the accumulation of these inorganic particles at the surface or inside the filters and possible maintenance strategies to extend filters useful life (e.g., washing the filtering matrix).

Table 5.9 shows the regular car wash and urban wastewater values for certain parameters (Zaneti *et al.*, 2012).

	COD (mg/L)	Surfactants (mg/L)	Total phosphorus (mg/L)	Total nitrogen (mg/L)		
Urban wastewater	430	4	7	40		
Car wash wastewater	191	21	1	9		
Bus wash wastewater	307	6.3	8.5	5		
Truck wash wastewater	600	21	8.5	30		

Table 5.9. Urban and car wash wastewater characteristics (adapted from Zaneti et al., 2012)

Bibliographic data analytical results (Bhatti *et al.*, 2010; Zaneti *et al.*, 2011; Zaneti *et al.*, 2012 and Zaneti *et al.*, 2013) are very similar to those obtained in the water sampling characterisation at the inlet of the ST (Minaqua, 2014). However, if we compare bibliographic data with the water arriving to the pilots the values are lower in the pilot's

influent, especially in organic matter and solids. This is probably because in the mentioned studies, samples were taken before any pretreatment without settling. It must be also pointed out that the values included in the literature (Zaneti *et al.* 2011) vary greatly, which shows the importance of obtaining a large number of samples to be able to draw reliable conclusions.

Regarding nutrients, concentration in nitrogen and phosphate forms in the influent was low and similar to the literature. The concentration of the three types of surfactants analysed was lower than expected (only non-ionic surfactants and in low concentration were found: maximum 0.6 mg/L), which may be due to its rapid biodegradability, high dilution and an optimized detergent dosing. Other wastewater characterisations were additionally carried out in parallel in several car washing facilities in the same project (Minaqua, 2014), and presented surfactant concentrations slightly larger: 2.6 mg/L of non-ionic surfactants (these values are from a car wash facility in Girona managed by the same company as the Montfullà facility). The average of 15 sampling campaigns conducted in another car wash facility in the Basque Country (within the same project) gave a concentration of 0.25 mg/L for non-ionic surfactants and bdl for anionic surfactants.

The hydrocarbons, oils and fats parameter values were low (average 0.2 mg/L and maximum 0.3 mg/L). Again these results are different from those of the literature (oil and fat values close to 5 mg/L), and from the data obtained from initial characterisation at the inlet of the ST (hydrocarbons average of 2.1 mg/L and oils and fats average of 13.9 mg/L). These results can be explained because these components are less dense and float so they move to the second ST. Therefore, it can be estimated that a significant part of these components is not injected by the pump into the pilots.

In fact, the initial water characterisation results (Minaqua, 2014) showed values before the ST of 1-4 mg/L of hydrocarbons and 12-15 mg/L of fats, and at the outlet of the third ST (before the hydrocarbon separator) values of 12-14 mg/L and 1-2 mg/L of hydrocarbons. Therefore only about 10% of the fats and oils from the initial effluent were sent to the pilots.

In terms of microbiological parameters, pH, conductivity and most soluble substances (chlorides, sulphates, calcium and magnesium) results are in accordance with bibliographical data (Bhatti *et al.*, 2010; Zaneti *et al.*, 2011; Zaneti *et al.*, 2012).

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The basic purpose of the pilot plants is to generate treated wastewater of enough quality to be recycled for car wash facilities (in the most appropriate arches). The purpose is to recycle the wastewater treated by the pilots will for the same purpose in the same facility. However, there is no mandatory regulation for recycling. There are some recommendations from companies (for internal use) on the quality that the water must have (tap water or recycled) to be used in car washing equipment.

Since there is no legal regulation setting the specific limit for most of the recycling parameters, this study has taken as one of the quality targets the values included in Royal Decree 1620/2007 "establishing the legal regime for reusing treated water." In fact, Annex IA of this RD shows the quality criteria for reusing water according to different uses. This annex indicates the quality required for 1. Urban use/Quality 1.2/Services/ d) Industrial vehicle washing. Table 5.10 shows the quality criteria for urban wastewater reuse, quality 1.2 Services (including industrial vehicle washing). These values allow comparing the water quality obtained in the study with the decree values and ensure that the water to be recycled does not pose a health or chemical risk.

	Maximum acceptable value					
Water use	Intestinal nematodes	E. coli	SS	Turbidity	Other criteria	
1.2. Quality Services						
a) Urban green areas watering						
b) Street washing		200			Legionella spp.	
c) Fire-fighting systems	1 egg/10L	CFU/100mL	20 mg/L	10 NTU	(if there is risk of aerosolization)	
d) Industrial vehicle washing						

Table 5.10. Quali	ty required according	to RD 1620/2007	for urban use qual	ity 1.2. Services
		<i>,</i>		1

5.4.2. Performance of the pilots

5.4.2.1. Pilot efficiency: inlet quality and removal efficiency

5.4.2.1.1. Horizontal subsurface flow constructed wetland

Tables 5.11, 5.12 and 5.13 show the overall water quality results for the HFCW influent and effluent and pollutant removal efficiency (for all applied HLs).

Parameters	Units	Average	Max	Min	SD	dl	% <dl< th=""></dl<>
рН		7.4	7.9	6.4	0.3	0	0.0
Redox	mV	-1.14	170	-90.4	58	±2000	0.0
EC	µS/cm	486	759	306	76.4	0	0.0
DO	%	15.3	85.9	0.3	19.7	-	2.6
DO	mg/L	1.3	6.7	0.0	1.5	0	0.0
Turbidity	FNU	2.8	11.5	0.1	3.0	0	2.7
COD	mg/L	14.5	36.0	bdl	11.5	10	40.1
dOOp	mg/L	bdl	24.0	bdl	8.2	10	65.7
pCOD	mg/L	6.1	15.0	0	5.3	-	-
BOD ₅	mg/L	bdl	15.0	bdl	3.8	5	67.5
SS	mg/L	4.7	29.0	bdl	9.0	3	64.7
VSS	mg/L	bdl	5.0	bdl	0.9	3	93.8
TKN	mg/L	bdl	3.5	bdl	0.7	3	87.5
N-NO ₃ ⁻	mg/L	0.6	2.3	bdl	0.6	0.5	62.5
N-NH ₄ ⁺	mg/L	bdl	0.3	bdl	0.1	0.1	81.3
P-PO4 3-	mg/L	bdl	0.4	bdl	0.1	0.1	87.5
S-SO ₄ ²⁻	mg/L	37.7	51.9	28.4	6.6	5	0.0
Cľ	mg/L	29.0	44.8	23.1	7.1	10	0.0
Ca ²⁺	mg/L	51.0	60.2	42.6	6.2	2	0.0
Mg ²⁺	mg/L	9.6	11.7	7.8	1.3	2	0.0
Alkalinity	mg/L CaCO₃	162.9	202.0	110.1	25.8	5	0.0
Anionic surfactants	mg/L	bdl	bdl	bdl	0.0	0.1	100.0
Cationic surfactants	mg/L	bdl	bdl	bdl	0.0	0.2	100.0
Non-ionic surfactants	mg/L	bdl	bdl	bdl	0.1	0.5	100.0
Hydrocarbons, oil and fats	mg/L	bdl	0.1	bdl	0.1	0.1	66.7
E. coli	CFU/100 mL	48	300	0	93	-	-
Legionella spp.	CFU/L	0	0	0	0	-	-

Table 5.11. HFCW effluent quality

Max=maximum, Min=minimum, SD=standard deviation, dl= detection limit, %<dl= percentage below dl

Parameter	% Removal Parameter (Average)		% Removal (Average)
Turbidity	96.7	P-PO4 ³⁻	100**
COD	73.3	S-SO4 ²⁻	16.7
dCOD	72.2	Cl	50.0
pCOD	70.8	Ca ²⁺	6.0
BOD ₅	81.3	Mg ²⁺	-3.2*
SS	88.5	Alkalinity	-12.1*
VSS	88.8	Anionic surfactants	***
TKN	100**	Cationic surfactants	***
N-NO ₃ ⁻	80.1	Non-ionic surfactants	100**
N-NH4 ⁺	100**	Hydrocarbons, oil and fats	75.2

Table 5.12. Removal of physicochemical pollutants in the HFCW (%)

*Variation, ** the average outlet values were bdl, *** the average inlet and outlet values were bdl

Parameter	Ulog Removal (Average)
E. coli	1.4
<i>Legionella</i> spp.	Absent

Table 5.13. Removal of microbiological pollutants in the HFCW (Ulog)

As for the organic matter parameters, very high performances were also obtained (especially with BOD₅), offering an effluent with very low concentrations for these parameters. The nutrients were eliminated or transformed in a distinct way depending on the parameter: the few nitrates entering the HFCW were almost entirely eliminated by plant absorption mechanisms and/or via denitrification and volatilization (Vymazal and Kröpfelová, 2009). Phosphates they were completely removed. The phosphate elimination processes in HFCWs are plant absorption and absorption/precipitation (Vymazal, 2007). However, concentrations of these nutrients were very low in the inlet; therefore they were almost completely absorbed by plants. This is proved by the plant growth (see Figure 5.9).



Figure 5.9. State of Phragmites australis in the HFCW

As seen in Figure 5.9, the plants in the area closest to the inlet have considerably greater growth than those in the outlet area, with a gradual descent. This may be explained by the fact that the few nutrients found in the inlet wastewater were absorbed by the plants, therefore these nutrients are gradually depleted as the water circuit progresses. TKN and ammonia removal were almost 100%, but it is important to once again point out the low inlet concentrations. The soluble forms, such as the calcium, magnesium, sulphate and alkalinity did not vary substantially. The salinity did not show considerable variations, even over the summer months when evapotranspiration was higher. To make conclusions regarding this data, it is necessary to wait for a second year of monitoring, since the plants will be more developed and therefore the evapotranspiration may increase, leading to an increase in salinity.

The anionic and cationic surfactants were bdl in the inlet and outlet of the HFCWs. For the non-ionic detergents, they were always found to be bdl in HFCWs outlet. Removal of non-ionic surfactants from municipal wastewater using HFCW was studied for Sima and Holcova (2011). Non-ionic surfactants were removed with a high efficiency reaching 99.1%. The study found that non-ionic surfactants were degraded both under aerobic and anaerobic conditions. However, because of the low concentrations of surfactants in our study, it was not possible to go in depth in the study of degradation/removal of surfactants in HFCWs.

Hydrocarbons, oils and fats were almost entirely eliminated in the HFCW, with only one sample having values over the detection limit, with a value of 0.1 mg/L. However it must be noted that the influent concentrations were very low and that almost all oils and fats accumulated in the HFCW inlet area and clogged it (section 5.4.2.5). Polyaromatic hydrocarbons can be eliminated in CWs by various mechanisms

(Vymazal, 2014; Xu *et al., 2015*). However, as well as for the detergents, in our study it was not possible to go deep in the study of the removal of these compounds in the pilots due to the low concentrations. Regarding alues of dissolved oxygen and the redox potential in the HFCW effluent, it was observed that the concentration of oxygen decreased gradually in the HFCW and that the redox potential had slightly negative values (-1), indicating that the HFCW presented aerobic as well as anoxic/anaerobic areas.

Finally, regarding microbiological parameters, there was a good elimination of *E. coli*, with an average of elimination of 1.4 Ulog, a value that is quite characteristic of these systems (between 1-2 Ulog) (Huertas, 2009; Torrens *et al.*, 2010; Sasa, 2014). In Table 5.14, the HFCW pilot effluent values with those established by RD 1620/2007 are compared.

 Table 5.14. Comparison of HFCW pilot effluent quality with the established on the RD

 1620/2007

	Nematodes E. coli		SS	Turbidity	Legionella spp.
	Eggs/L	CFU/100mL	mg/L	FNU	UFC/L
RD Value	1	200	20	10	100
% > RD	0	12	0	5.4	0

RD 1620/2007 is very strict in regards to the self-management plans. These selfmanagement plans include a minimum number of samples for the parameters indicated in each use. In this study there has not been strict compliance with this number of samples, therefore the % > RD was calculated taking into account the number of analysis of our study. In Annex I.C of RD 1620/2007, it is established that 90 % of the samples may not exceed the established values. Furthermore, it is indicated that the samples may not exceed certain maximum established thresholds (Maximum Allowable Value): 1 Ulog in the case of *E. coli* and *Legionella* spp.; 50 % for SS and 100% for turbidity and intestinal nematodes.

In the HFCW effluent, for turbidity, only 5.4 % of the samples presented higher values, with these values being lower than 20 FNU, thus complying with the criteria for this parameter. For *E. coli*, 12% of the samples had values exceeding 200 CFU/100mL, with the maximum allowed deviation being 10 % of the samples. These results indicate that, overall, the HFCW effluent may be used for recycling purposes in the carwash without health risk problems. But for greater safety, a final chlorination is recommended after the HFCW. A final chlorination would permit to have residual chlorine which will

act as a disinfectant in the pipes; thereby preventing *E. coli* recontamination or growths. It would also serve to control *Legionella* spp.

As explained in the material and methods section the HFCW has two piezometers (PIEZ.1 and PIEZ.2 at 4.60 and 12.60 m from the inlet). The results of the samples analysed in these two points are shown in Table 5.15.

Parameters	Units	Average Inlet	Average PIEZ.1	Average PIEZ.2	Average HFCW Outlet
pН		87.9	7.3	7.3	7.4
Redox	mV	88.4	-405.6*	-24.0*	-1.14*
EC	µS/cm	503.2	486.9	48.1	486
DO	%	14.5	11.8	14.2	15.3
DO	mg/L	1.2	1.2	1.25	1.3
Turbidity	FNU	85	18.7	6.03	2.8
COD	mg/L	48.8	26.2	20.1	13
dCOD	mg/L	22.3	15.9	13.5	6.2
pCOD	mg/L	30.8	10.3	9.1	9.0
BOD ₅	mg/L	14	9	5.7	2.6
SS	mg/L	41	7.8	8.1	4.7
E. coli	CFU/100mL	1262	296	76	48

Table 5.15. Water quality in the piezometers of the HFCW

In Table 5.15, a progressive increase in quality (lower concentrations) can be observed in organic matter parameters, turbidity and *E. coli*. However, there were no considerable variations in SS for the first piezometer as compared to the second and the final effluent. This may be due to the fact that the SS are essentially removed by mechanical sedimentation and filtration processes in the HFCW (UN-HABITAT, 2008), and these mechanisms are not influenced by the HRT. Furthermore, a change in redox potential was observed within the HFCW as approaching the outlet: the results become more positive, that is, the filter becomes less anaerobic.

5.4.2.1.2. Vertical flow constructed wetland

Tables 5.16, 5.17 and 5.18 show all the results of the outlet water quality and the pollutants removal in the VFCW for all the applied HLs.

Parameters	Units	Average	Max.	Min.	SD	dl	% <dl< th=""></dl<>
рН		7.5	8.0	7.0	0.2	0	0.0
Redox	mV	119.0	-7.9	-57.0	67.4	±2000	0.0
EC	μS/cm	537.1	285.1	-10.4	69.7	0	0.0
DO	%	47.8	784.0	393.7	16.2	-	0.0
DO	mg/L	4.5	78.7	25.5	1.6	0	0.0
Turbidity	FNU	16.2	8.4	2.2	17.7	0	0.0
COD	mg/L	10.5	68.0	bdl	10.5	10	55.0
dCOD	mg/L	bdl	12.0	bdl	1.6	10	92.5
₽COD	mg/L	5.1	58.0	-	10.0	-	0.0
BOD₅	mg/L	bdl	7.0	bdl	1.0	5	92.5
SS	mg/L	11.7	59.0	bdl	15.0	3	35.0
VSS	mg/L	bdl	6.0	bdl	1.5	3	84.2
TKN	mg/L	bdl	3.4	bdl	0.4	3	94.7
N-NO ₃ ⁻	mg/L	3.9	10.7	bdl	3.0	0.5	11.1
N-NH4 ⁺	mg/L	bdl	0.3	bdl	0.1	0.1	97.6
P-PO4 ³⁻	mg/L	bdl	0.6	bdl	0.1	0.1	94.1
S-SO4 ²⁻	mg/L	44.7	100.7	35.5	20.8	5	0.0
CI	mg/L	35.1	41.8	21.4	6.3	10	0.0
Ca ²⁺	mg/L	55.4	61.1	49.5	3.7	2	0.0
Mg ²⁺	mg/L	8.3	9.1	7.6	0.5	2	0.0
Alkalinity	mg/L CaCO ₃	153.1	176.4	128.1	16.1	5	0.0
Anionic surfactants	mg/L	bdl	bdl	bdl	0.0	0.1	100.0
Cationic surfactants	mg/L	bdl	bdl	bdl	0.0	0.2	100.0
Non-ionic surfactants	mg/L	bdl	bdl	bdl	0.0	0.5	100.0
Hydrocarbons, oil and fats	mg/L	bdl	bdl	bdl	0.0	0.1	100.0
E. coli	CFU/100 mL	9	90	0	19	-	-
Legionella spp.	CFU/L	240	1200	0	0	-	-

 Table 5.16.
 VFCW effluent quality

Max=maximum, Min=minimum, SD=standard deviation, dl= detection limit, %< dl= percentage below dl

Parameter	% Removal (Average)	Parameter	% Removal (Average)
Turbidity	80.9	P-PO4 ³⁻	100**
COD	78.5	S-SO4 ²⁻	13.1
dCOD	75.7	CI	39.5
pCOD	76.3	Ca ²⁺	-2.1*
BOD ₅	80.3	Mg ²⁺	5.7
SS	71.5	Alkalinity	-9.0*
VSS	86.3	Anionic surfactants	***
TKN	100**	Cationic surfactants	***
N-NO ₃ ⁻	-56.9*	Non-ionic surfactants	100**
$N-NH_4^+$	100** Hydrocarbons, oils and fats		100**

Table 5.17. Removal of physicochemical pollutants in the VFCW (%)

*Variation, ** the average outlet values were bdl, *** the average inlet and outlet values were bdl

Table 5.18. Removal of microbiological pollutants in the HFCW (Ulog)

Parameter	Ulog Removal (Average)	
E. coli	2.1	
Legionella spp.	Absent in inlet	

As seen in Tables 5.16, 5.17 and 5.18 the VFCW performed efficiently, offering a very good effluent quality for the physicochemical and microbiological parameters. The efficiency was high, particularly in regards to the COD and BOD₅. In terms of SS and turbidity, the outlet qualities were good, although variable, and the average turbidity was 16 FNU. The most important SS elimination mechanism in VFCW is filtration (Torrens *et al.*, 2009a). The % SS removal was about 75 %. Although this value is important, the VFCW was designed to create a biosolids surface layer. This layer of organic solids can reduce the infiltration velocities and increase filtration, leading to a larger retention of particulate substances (see section 4.4.2.6.).

In the case of the Montfullà pilot plant, in the VFCW, the water received from the car washing facility had few organic solids, thus the layer forming on the surface was quite thin. This leads to a faster infiltration and lower filtration. Across the same batch, the VFCW effluent turbidity has been observed to vary depending on the specific time of sampling (see Table 5.19). There is a part of the water that exits after a few minutes (as shall be explained in the section on hydraulic monitoring: 5.4.2.2) that presents higher turbidity and SS concentration.

Sampling time	Turbidity (FNU)
Prior to batch (small outflow)	2.6
Immediately following batch (very high outflow)	24.9
30 minutes after batch (average outflow)	7.3

Table 5.19. Turbidity changes based on time of sampling (HL=36cm/day, 4 bactches/day)

During the first year of operation, there have been no signs of filter clogging, even with HLs of 36 cm/day. Regarding organic matter parameters, the results (% removals) were also quite high, between 70 and 80 %. Nitrates were higher in the outlet of the VFCW as compared to the inlet, due to the fact that the system is aerobic and oxidises the ammonia into nitrates (Molle *et al.*, 2006). The low TKN, ammonia and phosphate loads entering the VFCW were completely eliminated. Unlike the HFCW, the plants developed similarly across the VFCW filter (given that the distribution of water on the surface is similar across the points of the filter). Therefore the plants may absorb nutrients in a similar way across the entire bed (see Figure 5.10).



Figure 5.10. State of *Phragmites* in the VFCW

The soluble ions, such as the calcium, magnesium, sulphate cations or alkalinity did not varied considerably during the treatment. Salinity also showed no significant variations. The anionic and cationic surfactants were bdl in the VFCW influents and effluents. As for the non-ionic surfactants, the elimination reached 100% (it should be noted that on very few occasions the influent values were greater than the thedetection limits values). Non-nionic surfactants can be degraded both under aerobic and anaerobic conditions in CWs (Sima and Holcova, 2011). Therefore these surfactants could be removed and/or transformed in VFCWs. Because of the low concentrations of surfactants on VFCW influent and effluent, it was not possible to go deeper into the study of degradation/removal of surfactants in VFCWs. Hydrocarbons, oils and fats were removed in the pilot. Once again, it should be noted that the inlet concentrations are very low. Polycyclic aromatic hydrocarbons can be eliminated in VFCWs (Vymazal, 2014). However in our study it was not possible to go into detail in the study of the removal of these compounds in the pilots due to their low concentrations.

Unlike the HFCW where the formation of oily looking deposits was observed in the inlet area; in the VFCW filter no major accumulations have been observed which may lead to clogging. When observing the outlet values and results of dissolved oxygen elimination and redox potential, it was found that the concentration of oxygen increased considerably and the redox potential had slightly greater positive values than in the inlet. These results indicate that the VFCW is an aerobic system that oxygenates the influent. For the microbiological parameters, there was a high E. coli elimination, with an average of 2.4 Ulog. These values are slightly greater than those normally observed in the VFCW which tend to range between 1 and 2 Ulog (Torrens et al., 2009b). However in the studied VFCW the filtration media was deeper (100 cm). Removals greater than 2 Ulogs for *E. coli or* fecal coliforms has been observed in IP systems with 150 cm of sand (Folch, 1999; Brissaud et al., 2007). Legionella spp. was always absent in the inlet, and presenented one positive in the outlet of the VFCW, with a value of 1200 UFC/L. When comparing with the RD 1620/2007, this value represents 3 Ulog, and therefore it is within the range of the maximum acceptable threshold as indicated in Annex 1C of the RD. Despite this, the feeding system was disinfected with chlorine and the sampling was repeated after 15 days, with negative results at the outlet of the VFCW and in several points of the car wash facility. Table 5.20 compares the effluent values of the VFCW pilot with those from the RD 1620/2007.

	Nematodes	E. coli	SS	Turbidity	Legionella spp.		
	Eggs/L	CFU/100mL	mg/L	FNU	CFU/L		
RD Value	1	200	20	10	100		
% > RD	0	0	15	43	20		

Table 5.20. Comparison of the VFCW outlet quality with that of RD 1620/2007

For turbidity, 43% of the samples had higher values than indicated in the RD. Although the values are very close to 10 (the average is 15), they do not comply with the regulations in regards to this parameter. As for the SS, turbidity do not always comply with the regulations (15% of the values exceed the limits). Follow-up of the VFCW performances for a longer time period would be necessary. The organic layer would increase with time, thereby increasing filtration and providing higher percentage removal of suspended solids and turbidity. For *E. coli*, the results are always lower than the value of 200 CFU/100mL established by RD 1620/2007. As for *Legionella* spp., it

also fails to comply with the thresholds, in one of the five samples there was a value of 1200 CFU/L. These results indicate that it would be useful to add a final disinfection treatment (such as chlorination). This chlorination would also provide residual chlorine which would serve to disinfect the pipes thereby preventing pathogens recontamination.

5.4.2.1.3. Infiltration-percolation pilot

The following sections show the results of the IP pilot (DF and IP). The DFs function as pretreatment for the IP, in order to eliminate the largest solids for prevent clogging problems of the irrigation system that feeds the IP. Table 5.21 shows the results of the outlet water quality of the disk filters and its removal efficiency.

Parameters	Units	Average	Max	Min	Removal
Temperature	°C	19.7	28.4	8.0	-
рН	pH units	7.8	9.0	7.2	-
Redox	mV	81.5	223.3	-112.6	-
EC	μS/cm	506.5	1045	373.7	-0.6 %*
DO	%	10.9	42.9	0.0	21.4 %
DO	mg/L	1.1	5.1	0.0	8.3 %
Turbidity	FNU	59.3	199	0.5	30.6 %
COD	mg/L	39.8	120.0	10.0	18.3 %
dCOD	mg/L	21.5	98.0	10.0	21.5 %
pCOD	mg/L	18.3	53.0	10.0	4.5 %
BOD ₅	mg/L	12.3	46.0	5.0	14.3 %
SS	mg/L	19.7	46.0	4.0	51.2 %
VSS	mg/L	6.4	12.0	3.0	57.3 %
TKN	mg/L	4.3	6.8	3.0	2.4 %
N-NO ₃ ⁻	mg/L	0.3	0.5	0.2	0.0 %
N-NH4 ⁺	mg/L	0.05	0.05	0.05	0.0 %
P-PO4 ³⁻	mg/L	0.05	0.05	0.05	0.0 %
Alkalinity	mg/L CaCO $_3$	157.1	178.9	128.5	-4.1 %*
Anionic surfactants	mg/L	bdl	0.6	0.1	**
Cationic surfactants	mg/L	bdl	0.2	0.2	**
Non-ionic surfactants	mg/L	bdl	1.9	bdl	7.2 %
E. coli	CFU/100mL	620	4000	0	0.31 Ulog

Table 5.21. Outlet quality and removal (% or Ulog) for the DF

Max=maximum, Min=minimum, * Variation, ** the average inlet and outlet values were bdl

DFs removed particulate matter. Soluble components (dissolved COD, nitrates, ammonia, phosphates, alkalinity, detergents) presented similar values in the DFs' inlet.

DFs removed approximately half of the suspended solids (51 % for the SS and 57 % for the VSS). Turbidity was reduced by 30%. The total COD was reduced by approximately 20%. As seen in Table 5.19, COD removed the particulated COD: the soluble particles are not substantially modified (< 5%). These results were expected (Alcalde *et al.*, 2007) given that filtration is the mechanism of the disk filters. *E. coli* presented a small reduction of 0.31 Ulog. This removal could be explained due to the retention of *E. coli* associated with the SS. Tables 5.22, 5.23 and 5.24 show the overall results of the outlet water quality and the pollutant removals in the IP for all of the applied HLs.

Parameters	Units	Average	Max.	Min.	SD	DL	% <dl< th=""></dl<>
рН		7.8	9.4	6.5	0.5	0	0.0
Redox	mV	126.0	575.9	-58.0	100	±2000	0.0
EC	µS/cm	525.8	765.0	356.0	79.1	0	0.0
DO	%	53.1	93.3	0.5	20.0	-	0.0
DO	mg/L	4.7	10.0	0.0	2.1	0	0.0
Turbidity	FNU	1.1	5.1	0.0	1.4	0	0.0
COD	mg/L	bdl	16.0	bdl	4.6	10	48.6
dCOD	mg/L	bdl	15.0	bdl	2.4	10	89.2
pCOD	mg/L	5.7	11.0	5.0	3.8	-	-
BOD₅	mg/L	bdl	9.0	bdl	1.2	5	94.6
SS	mg/L	bdl	6.0	bdl	0.8	3	97.2
VSS	mg/L	bdl	bdl	bdl	0.0	3	100
TKN	mg/L	bdl	bdl	bdl	0.0	3	100
N-NO ₃ ⁻	mg/L	2.7	2.8	bdl	0.7	0.5	0.0
$N-NH_4^+$	mg/L	bdl	0.3	bdl	0.1	0.1	93.7
P-PO4 ³⁻	mg/L	bdl	0.5	bdl	0.1	0.1	93.4
S-SO4 ²⁻	mg/L	41.3	52.0	32.2	6.4	5	0.0
Cl	mg/L	42.3	64.8	22.4	13.1	10	0.0
Ca ²⁺	mg/L	50.2	56.7	43.0	5.2	2	0.0
Mg ²⁺	mg/L	10.0	12.6	7.0	2.0	2	0.0
Alkalinity	mg/L CaCO₃	157.2	185.6	116.2	18.6	5	0.0
Anionic surfactants	mg/L	bdl	bdl	bdl	0.0	0.1	100
Cationic surfactants	mg/L	bdl	bdl	bdl	0.0	0.2	100
Non-ionic surfactants	mg/L	bdl	bdl	bdl	0.0	0.5	100
Hydrocarbons, oils and fats	mg/L	bdl	bdl	bdl	0.0	0.1	100
E. coli	CFU/100 mL	4	180	0	30	-	-
Legionella spp.	CFU/L	0	0	0	0	-	-

Table 5.22. IP effluent quality

Max=maximum, Min=minimum, SD=standard deviation, dl=detection limit, %< dl= percentage below dl

Parameter	% Removal (Average)			Parameter	% Removal (Average)		
	DF	IP	DF+IP		DF	IP	DF+IP
Turbidity	30.6	68.1	98.7	P-PO4 ³⁻	0	100**	100**
COD	18.3	62.5	80.8	S-SO4 ²⁻	nd	nd	19.7
dCOD	21.5	55.9	74.2	Cl	nd	nd	27
_p COD	4.5	68.7	90.2	Ca ²⁺	nd	nd	7.5
BOD_5	14.3	82.9	87.4	Mg ²⁺	nd	nd	-12.3*
SS	51.2	81.7	96	Alkalinity	-4.1*	-11.8*	-15.9*
VSS	57.3	39.1	90.3	Anionic surfactants	***	***	***
TKN	2.4	100**	100**	Cationic surfactants	***	***	***
$N-NH_4^+$	0	100**	100**	Hydrocarbons, oils and fats	nd	100**	100**

Table 5.23. Removal of physicochemical pollutants in DF, IP and DF + IP (%)

*Variation, **the average outlet values were bdl, *** the average inlet and outlet values were bdl

Table 5.24. Remova	l o	ⁱ microbiologica	l pollutants i	in the pilot DF+IP	(Ulog)
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Parameter	Ulog Removal (Average)					
	DF	IP	DF+IP			
E. coli	0.3	2.1	2.4			
Legionella spp.	Absent	Absent	Absent			

As seen in Tables 5.22, 5.23 and 5.24, the DF+IP combination performed very efficiently, offering an optimal effluent quality for the physicochemical and microbiological parameters. The IP outlet had a maximum quality effluent with values that are almost always bdl for the majority of the parameters (see Table 22, column %>dl). Furthermore, the IP obtained a very consistent quality with very low SD and similar maximums and minimums. These results suggest the capacity of the IP system to adapt to flow and load variations without compromising the effluent quality. It should be noted, as mentioned in the previous section, that the disk filters remove a considerable part (approx. 50 %) of the solids. This helps to prevent the clogging of the irrigation system and the sand, thereby improving the functioning of the performances of the IP system.

The IP treatment performance was very high, particularly in regards to organic matter, suspended solids, turbidity and *E. coli.* In the IP outlet, the SS were, in 98 % of the cases, bdl, being 100% bdl for volatile suspended solids (organic solids). The average total COD of the outlet was lower than 10 mg/L and BOD₅ less than 5 mg/L. The

removal mechanisms in IP are mostly filtration and oxidation (Brissaud *et al.*, 2007). The IP matrix consists of fine sand, which offers, along with the efficient distribution of water via the irrigation system, quite high purification results, even for the HL of 36 cm/day. These results are in agreement with those of the bibliography (Brissaud *et al.*, 2007; Huertas *et al.*, 2007). During the first year, no signs of clogging appeared in the filter matrix, even with the highest HL of 36 cm/day. However, the irrigation system clogged (see section 5.4.2.5.) after 10 months of functioning at a HL of 36 cm/day, despite the good functioning of the disk filters and the low values of SS and COD in the IP inlet (SS \approx 20 mg/L, COD \approx 40 mg/L). This clogging is produced by an accumulation of particles having a viscous appearance, similar to the deposits that accumulated in the inlet gravel of the HFCW. The irrigation network that was initially placed did not include a purge system; therefore, as explained in the operating section, this irrigation system was changed for another having a purge system.

Nitrates were slightly greater in the IP outlet than in the inlet, since the system functions aerobically and transforms (oxidises) the ammonia into nitrates (Brissaud et al., 2007; Huertas et al., 2007). The elimination of TKN and ammonia was almost complete: in the IP outlet, practically 100% of the samples were bld. The same occurred with the phosphates. Once again, the very low nutrient inlet values should be noted. The soluble forms, such as the calcium, magnesium and sulphate cations or alkalinity did not vary significantly. There were no considerable variations in salinity, suggesting that evaporation or evapotranspiration via the grass is almost non-existent. The anionic and cationic surfactants were bdl in the IP inlet and outlet. As for the nonionic surfactants, elimination was found to be 100% (it should be noted once more that on few occasions inlet values have been greater than the detection limit). Hydrocarbons, oils and fats were completely eliminated in the pilot, always being found to be bdl. However, once again it is to note that the inlet values of this parameter were quite low. When observing the effluent values and those of dissolved oxygen and redox potential elimination, it can be seen that the concentration of oxygen increases substantially as it passes through the pilot IP and that the redox potential also had greater positive values than in the inlet. These results indicate that the IP functions correctly in an aerobic way, and oxygenates the influent.

For the microbiological parameters, there was a very high elimination of *E. coli*, with an average removal of 2.4 Ulog (0.3 Ulog for the DF and 2.1 for the IP). The value of 2.1 Ulog for the IP is characteristic of the IP systems having similar HLs (Huertas *et al.,* 2007; Torrens *et al.,* 2009b). Regarding *Legionella* spp., it was absent in both the inlet

and the outlet. In Table 5.25, the values of the IP pilot effluent are compared with those established by the RD 1620/2007.

	•		-		
	Nematodes	E. coli	SS	Turbidity	Legionella spp.
	Eggs/L	CFU/100mL	mg/L	FNU	CFU/L
RD Value	1	200	20	10	100
% > RD	0	0	0	0	0

Table 5.25. Comparison of IP pilot effluent quality with the established on the RD 1620/2007

Table 25 shows how, for all the parameters, there is a 100% compliance with the quality limit values established by RD 1620/2007. These results indicate that the DF+IP combination produced an effluent having optimal quality for being recycled.

5.4.2.2. Hydraulics

5.4.2.2.1. Horizontal subsurface flow constructed wetland

Table 5.26 presents the results of the monitoring of the inlet and outlet flows of the HFCW. For each period, a hydraulic monitoring of the inlet and outlet flow during the same day was carried out in order to verify any water loss caused by evapotranspiration.

			Period 1 HL1	Period 2 HL2	Period 3 HL3
Average		I	402	762	1393
	Flow (L/day)	0	370	716	1226

Table 5.26. Summary of flows in the inlet (I) and outlet (O) of the HFCW

As for the outlet flow, testing was conducted on three HLs (HL1=4 cm/day, HL2=7 cm/day and HL3=13 cm/day). A small amount of water was lost between the inlet and the outlet: 8% in period 1 (HL1), 6% in period 2 (HL2) and 12% in period 3 (HL3). These small losses may be due to evapotranspiration of water in the HFCWs due to the long HRTs. With the data from the first year of study, it may be concluded that the flow loss is approximately 10%. These water losses are to be expected based on the climate of the site and in the first year of operation due to the plant growth (Milani and Toscano, 2013).

It is recommended that, over the following years, more hydraulic effluent flow controls be conducted in order to gather additional data. It is especially recommended that testing to carry out tests during the months of the highest temperatures (July and August) when evapotranspiration tends to be the greatest. Figure 5.11 shows the instant flow profiles of the effluents in HFCW for the 3 HLs.



Figure 5.11. HFCW effluent flow rates (HL1, HL2 and HL3)

5.4.2.2.2. Vertical subsurface flow constructed wetland

Flow rates monitoring tests were conducted on the effluent. The inlet flow has been calculated based on the minutes of functioning of the pump that feeds the VFCW. The pump flow is 4.8 m³/hour. Table 5.27 shows a summary of the average inlet and outlet flows of the VFCW for each period.

			,	() ()	
			Period 1	Period 2	Period 3
			HL1	HL2	HL3
			4-8 batches/day	4-8 batches/day	4-8 batches/day
Average Flow (L/da	Flow (L/day)	Ι	402	762	1393
	Flow (L/uay)	0	37	716	1226

able 5.27. Summa	y of flows in	the inlet (I)	and outlet (O)	of the VFCW
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For small flows, the method used for balancing the inlet/outlet flow performed correctly, since flow rates are most consistent. For greater HLs, this methodology did not worked properly. For future outlet flow testing, it is recommended to take measurements every minute or seconds. However, this data provides an indication that the flow loss via evapotranspiration is low in VFCW. It should be noted that in the most reliable data

(period 1, HL1), losses are about 7%. These losses are to be expected based on the climate of the site and the plant growth of the VFCW plants which have lower evapotranspiration rates than the HFCWs. This is due to the lower water retention time in the vertical systems (days in the HFCWs as opposed to hours in the VFCWs). It is also recommended that additional testing be conducted on HFCW outlet flows. Figures 5.12, 5.13 and 5.14 show the evolution of flow rates for the three HLs. Figures 5.13 and 5.14 show that for each HL, there is changing of the hydraulic behaviour for the two application modes (4 and 8 batches per day).







Figure 5.13. VFCW effluent flow rates (HL 2: 4 and 8 batches)



Figure 5.14. VFCW effluent flow rates (HL 3: 4 and 8 batches)

Upon increasing the HL (Figures 5.13 and 5.14), the behavior changed radically: the flow peaks were much sharper and appeared between 15 and 30 minutes approximately. Between applications, the flow is virtually non-existent. This means that a considerable part of the water infiltrates and percolates more quickly in the filter (plug flow). When applying a larger quantity of water in the filter, the pressure exerted by the water is greater, leading to the percolation occurring faster. As it percolates more quickly, the outlet flow is virtually zero between applications, allowing for the filter to have less retained water (lower humidity). Thus, when applying a new batch the filter is drier (less humid), and the water percolates more quickly (the lower humidity within the filter the less resistance to the water filtration, and therefore a higher infiltration speed).

These results coincide with the results of other studies carried out on the VFCW (Molle *et al.*, 2006, Torrens *et al.*, 2009a). When comparing the two application modes (4 or 8 applications per day) for the same HL it is observed that with a greater number of applications, the maximum flow rates are lower, and the curves tend to be horizontal. This means that for a greater number of applications, the water percolates more slowly. This behavior is due to the fact that when there are a larger number of applications, the time between applications is lower, such that the filter retains more humidity. On the other hand, upon applying less water in each batch, the vertical pressure is lower. These two effects result in a slower infiltration and percolation rates, as observed in the studies of VFCW treating swine slurry (4.4.2.5.).

5.4.2.2.3. Infiltration-percolation

As with the other pilot plants, several control tests have been carried out. The inlet flow has been calculated based on the minutes of functioning of the pump that feeds the IP.

The total flow provided was of 239.2 L/h. Table 5.28 summarises the inlet and outlet flows of the IP for each period.

			Period 1 HL1	Period 2 HL2	Period 3 HL3
			4-8 batches/day	4-8 batches/day	4-8 batches/day
Average	Elow (L/dov)	Ι	478	1914	3764
Average Flow (L/day)	0	532	2055	3725	

Table 5.28. Summary of flows in the inlet (I) and outlet (O) of the VFCW

The inlet and outlet flows presented similar values, indicating that losses from evapotranspiration were nearly zero. It should be noted that the follow-up of the outlet flow was manual (with a measuring cylinder and stopwatch), and those of the inlet were based on the calculation of the pump flow, thus these small variations in balance are probably caused by the the accumulation of precision errors occurring in these calculations. Overall it can be concluded that there were no flow losses. This is explained, on one hand, by the fact that the water application is by subsurface irrigation systems and on the other hand, by the fact that there are no macrophytes as is the case in the CWs. Thus, when recycling water from an IP, it may be considered that practically 100% of the applied flow will be found in the outlet. Figures 5.15, 5.16 and 5.17 show the evolution of the flow rates for the three HLs. Figures 5.16 and 5.17 also show for each HL two application modes (4 and 8 batches per day).



Figure 5.15. IP effluent flow rates (HL1: 8 batches)



Figure 5.16. IP effluent flow rates (HL 2: 4 and 8 batches)



Figure 5.17. IP effluent flow rates (HL 3: 4 and 8 batches)

Figure 5.14 reveals that for the low HLs (period 1) and with 8 applications, the outlet flow is constant without flow peaks. This means that the water percolates very slowly. The increase in HL the IP, as in the case of the VFCW, was carried out increasing the minutes of water feeding (with the instant flow of the pump always remaining constant). By increasing the HL the behavior changed, as in the VFCW, although in the case of the IP, the change occurs very smoothly: flow peaks occur between 2-4 hours after the batch is applied. The flow rate that feeds the IP is much lower than the flow rate of the VFCW, thus the flow peaks are smaller in the IP effluent.

When comparing the two application modes (4 or 8 daily applications) for the same HL it is observed that, as occurred in the VFCW, with a greater number of applications, the maximum flows are lower and the curves are more horizontal in shape. For a same HL the flow rate peak appears later when applying 8 batches/day opposed to 4 batches/day. This means that, as with the VFCW, with a greater number of applications, the water percolates more slowly. Unlike the VFCW, the flow peaks appear later (between 2-4 hours, approximately). This suggests that the HRT in the IP is greater, influencing the removal of pollutants as shall be explained below.

5.4.2.3. Comparison of pilot's efficiency

Table 5.29 shows the average removal percentages (physicochemical parametters) for the three pilots.

Parameter	% Removal (Average)				
Falanielei	HFCW	VFCW	DF+IP		
Turbidity	96.7	80.9	98.7		
COD	73.3	78.5	80.8		
dOOb	72.2	75.7	74.2		
pCOD	70.8	76.3	90.2		
BOD ₅	81.3	80.3	87.4		
SS	88.5	71.5	96		
VSS	88.8	86.3	90.3		
TKN	100**	100**	100**		
N-NO ₃ ⁻	80.1	-56.9*	-4.5*		
$N-NH_4^+$	100	100	100		
P-PO4 ³⁻	100**	100**	100		
S-SO ₄ ²⁻	16.7	13.1	19.7		
Cl	49.7	39.5	27.3		
Ca ²⁺	6.3	-2.1*	7.5		
Mg ²⁺	-3.2*	5.7	-12.3*		
Alkalinity	-12.1*	-9.1*	-15.9		
Anionic surfactants	***	***	***		
Cationic surfactants	***	***	***		
Non-ionic surfactants	100**	100**	100**		
Hydrocarbons, oils and fats	75.2	100**	100**		

Table 5.29. Comparison of performances of the pilots for physicochemical parameters (%)

*Variation, ** the average outlet values were bdl, *** the average inlet and outlet values were bdl

Table 5.30 shows that the three pilot plants presented very high removal percentage for turbidity and for all of the organic matter and SS parameters. Even when considering that the HLs applied to the VFCW are greater than those of the HFCW, the VFCW performance is slightly greater than that of the HFCW. This may be due to the fact that the oxidation of the organic matter occurs more efficiently in the VFCW, through the application of bacthes at high flow rates. The vertical flow and operation method favors filter oxidation. The DA+IP combination results in almost complete elimination of the SS and turbidity. The DA+IP system is the pilot plant with the best treatment performance. The filtration capacity of the IP is high, since the particle size of the filter is fine, and the flow rate applied (lower than the VFCW) with a subruface irrigation system results in a good distribution of water and a slower percolation. The

aerobic functioning of the system via batches permits and efficient oxidation of the dissolved organic matter.

HFCW presented percentage removal about 88 % for SS. The second best system in regarding percantege removal for solids is the HFCW. Finally, we consider the VFCW: the lower degree of solids removal, as previously mentioned, is likely to be due to the faster water percolation occurring in the vertical filter and also because the sand particle size is larger than in the IP. Figure 5.18 presents a graphic comparing the average percentage removal of the three technologies for SS and turbidity.



Figure 5.18. Average removal efficiency (%) for SS and turbidity

The purpose of experimenting with a VFCW pilot plant using an intermittent feeding mode and coarser sand was to test if this type of technology could effectively remove pollutants by applying wastewater from the car wash with high solid concentrations (and high OLR and SS loading rates per m²), However, during the first year of operation, given that the SS concentration was not very high, the limit ability to accept high SS concentrations could not be tested. Better results are anticipated in next years of operation when a thicker organic layer would be formed on the top of the VFCW filter.

Figure 5.19 shows the evolution of the turbidity for the three pilots during the first year of study. It can be observed that the DF+IP system produces a consistently well treated effluent having the lowest values (< 5 FNU). The VFCW has an effluent with a less constant quality and with higher values for this parameter. It may also be seen that in the HFCW effluent there is a slight increase in turbidity over time, which may be related to the increased HL over the first year. The effect of HL on the performance of the pilot plants is analysed in greater detail in the following section.



Figure 5.19. Turbidity evolution for the three pilot effluents

The VFCW and IP nitrified the effluents. The HFCW, on the other hand, presented anaerobic conditions and therefore did not produce nitrates. The three pilots eliminated TKN and ammonia almost entirely (values quite always bdl). The same occurred with phosphates.

Regarding calcium, magnesium and sulphate ions, there were no major variations in any of the three pilots. No further variations were found for alkalinity, with a slight increase being found in the effluent from all three pilot plants.

The (non-ionic) surfactants from the influent of the pilot plants were completely eliminated (100%) by the three technologies, but the concentrations of these surfactants in the influent were quite low. The same results were found for oils, fats and hydrocarbons: they were eliminated very efficiently in the three pilot plants, however their influent concentrations were quite low. As for the microbiological parameters (Table 5.30 and Figure 5.20), once again the DF+IP combination presesented the greatest removal.

Parameter		Ulog Elimination (a	average)		
				DF+IP	
	HFCW	VFCW	DF	IP	Total DF+IP
E. coli	1.4	2.1	0.3	2.1	2.4
Legionella spp.	Absent	Absent in inlet and 1 positive in outlet		Absent	

 Table 5.30. Comparison of performances of the pilot plants for the microbiological parameters (Ulog)

The IP and VFCW presented an average reduction of 2.1 Ulog for *E. coli*. However, for HFCW it was 0.7 Ulog. HFCW presented lower Ulog removals even with lower HLs. The removal mechanisms for *E. coli* seem to be more effective in aerobic systems with finer particle media (sand) and high depths of the filtering media (\approx 100 cm sand) such as in the IP and the VFCW.



Figure 5.20. Average E. coli (Ulog) removal in the pilots

5.4.2.4. Effect of the operational parameters

The effect of the HL and dosing modes on the effluent quality of the pilots is presented in this section. For the HFCW, increasing HLs were applied, from \approx 1cm/day to \approx 13 cm/day. Below, in Table 5.31, the average values of the effluent are shown for the parameters monitored in each period.

		Period 1 HL ≈1 cm/day	Period 2 HL ≈7 cm/day	Period 3 HL ≈13 cm/day
Parameters	Units	Average	Average	Average
Redox	mV	75.0	-5.2	1.2
EC	μS/cm	502.8	495.2	469.8
DO	%	25.2	14.9	10.6
DO	mg/L	2.4	0.7	1.0
Turbidity	FNU	1.1	1.3	4.7
COD	mg/L	bdl	16.0	14.0
dOD	mg/L	bdl	10.4	10.2
_P COD	mg/L	1.9	9.5	4.7
BOD ₅	mg/L	bdl	4.4	4.9
SS	mg/L	bdl	3.6	bdl
VSS	mg/L	bdl	bdl	bdl
TKN	mg/L	bdl	bdl	bdl
N-NO ₃	mg/L	0.7	bdl	0.6
N-NH ₄ ⁺	mg/L	bdl	0.1	0.2
P-PO4 ³⁻	mg/L	bdl	bdl	bdl
S-SO4 ²⁻	mg/L	34.0	37.1	39.9
Cl	mg/L	24.2	26.9	33.0
Ca ²⁺	mg/L	42.7	56.4	51.1
Mg ²⁺	mg/L	11.6	9.6	8.5
Alkalinity	mg/L CaCO₃	172.9	157.8	149.6
Anionic surfactants	mg/L	bdl	bdl	bdl
Cationic surfactants	mg/L	bdl	bdl	bdl
Non-ionic surfactants	mg/L	bdl	bdl	bdl
Hydrocarbons, oils and fats	mg/L	bdl	bdl	bdl
E. coli	CFU/100mL	0	33	89
Legionella spp.	UFC/L	0	0	0

bdl=below the technique's detection limit

The effect of HL on the effluent concentrations for the HFCW may be observed in:

Organic matter parameters: an increase of the concentrations in the HFCW effluent was found for all of the organic matter parameters (especially in dCOD) when the HL increased from ≈1cm/day to ≈5 cm/day. The change of the HL from 5 cm/day to 13 cm/day do not led to changes in the quality of these parameters. The decrease in the removal of organic matter upon increasing the HL may be explained by the decreased HRT, resulting in a shorter degradation time for the organic matter.

- SS and turbidity: no major differences were found in these parameters, only turbidity when passing from HL2 (7 cm/day) to HL3 (13 cm/day), although they were not substantial. The removal mechanisms of these parameters are mainly sedimentation and filtration. These mechanisms are less affected by the decrease of the HRT.
- DO and redox: the increase in HL led to a decrease in oxygen concentration and the aerobic conditions of the system. The increase in HLs leads to a decrease in oxygen inside the filtering matrix. Figure 5.21 shows the changes occurring in redox potential over time with the increasing HL. There is a decrease in the redox potential with the increased of HL and thus organic loads over time.



Figure 5.21. Evolution of redox potential in the HFCW effluent

The other parameters were not affected by the increase in HL. The average values established by RD 1620/2007 (SS, turbidity, *E. coli* and *Legionella* spp.) were not exceeded in any of the HLs, even for HL3.

The effect of HL on the effluent quality in the VFCW may be seen in the slight increase of SS and turbidity (Table 5.32). There was also a slight decrease in dissolved oxygen observed as the hydraulic load increases. The increase in HL may result in an increased infiltration rate as there is a much larger water height of batch exerting greater pressure on the filter. This may lead to faster water flow particularly in the moment after the application of the batch, and to an increase in turbidity and SS. Thus the HL of 36 cm/day (HL3) is constraining for the VFCW. For the rest of the parameters (nutrients, organic matter, solids, ions, cations, detergents, hydrocarbons, oil and fat, *E*.

coli, Legionella spp.) no effect was observed in response to the increased HL. The fractionation of HL did not affect the VFCW effluent quality. The great number of values bdl maked difficult to observe the effect of operational parameters in the VFCW pilot.

		Period 1 HL ≈6 cm/dav		Perio HL ≈18	od 2 cm/day	Period 3 HL ≈36 cm/day	
Parameters		4 batches	8 batches	4 batches	8 batches	4 batches	8 batches
Redox	mV	130.4	124.5	51.2	83.9	171.5	77.2
EC	µS/cm	508.6	503.0	512.0	473.4	670.3	468.1
DO	mg/L	5.4	4.1	4.4	4.5	4.7	4.4
Turbidity	FNU	6.7	12.5	14.0	16.7	18.3	20.9
COD	mg/L	bdl	bdl	11.2	10.4	10.3	bdl
dCOD	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
₽COD	mg/L	0.0	4.0	4.8	5.4	4.8	2.5
BOD ₅	mg/L	bdl	bdl	4.1	bdl	bdl	bdl
SS	mg/L	3.4	4.2	5.3	10.2	18.6	13.3
VSS	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
TKN	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
N-NO ₃ ⁻	mg/L	6.5	6.4	1.6	0.6	3.8	3.0
$N-NH_4^+$	mg/L	bdl	bdl	bdl	bdl	0.21	bdl
P-PO4 ³⁻	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
S-SO4 ²⁻	mg/L	49.5	42.0	44.4	39.6	34.6	35.5
CI	mg/L	31.3	21.4	29.8	25.8	64.0	33.1
Ca ²⁺	mg/L	57.9	51.1	61.1	58.7	47.7	55.5
Mg ²⁺	mg/L	8.0	7.6	8.9	8.5	7.8	8.9
Alkalinity	mg/L CaCO₃	154.0	159.1	160.7	159.9	143.1	137.9
Anionic surfactants	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
Cationic surfactants	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
Non-ionic surfactants	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
Hydrocarbons, oils and fats	mg/L	nd	bdl	nd	bdl	bdl	bdl
E. coli	CFU/100 mL	0.0	0.0	0.6	16.0	14.0	2.0
Legionella spp.	CFU/L	nd	0	nd	0	1200	0

Table 5.32. VFCW average efffluent quality for three HLs

bdl=below detection limit, nd=not determined

Below, in Table 5.33, the IP average effluent values are shown for the three tested HLs.

		Period 1		Period 2		Period 3	
Parameters		n∟ ≈5 c 4 batches	8 batches	IL ≈10 4 batches	8 batches	4 batches	8 batches
Redox	mV	130.4	124.5	51.2	83.9	171.5	77.2
EC	µS/cm	479.6	535.6	491.8	481.8	637.9	464.3
DO	mg/L	4.8	4.5	4.5	3.8	6.0	3.4
Turbidity	FNU	2.0	0.2	0.6	0.4	2.0	1.2
COD	mg/L	bdl	11.8	12.8	bdl	bdl	bdl
dCOD	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
	mg/L	0.0	3.8	4.8	2.5	2.5	2.5
BOD ₅	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
SS	ma/L	bdl	bdl	bdl	bdl	bdl	bdl
VSS	ma/L	bdl	bdl	bdl	bdl	bdl	bdl
TKN	ma/L	bdl	bdl	bdl	bdl	bdl	bdl
N-NO ₃ ⁻	ma/L	1.9	0.9	2.1	0.2	1.9	1.25
N-NH₄ ⁺	ma/l	bdl	bdl	bdl	bdl	bdl	bdl
P-PQ ³⁻	mg/⊑	bdl	bdl	bdl	bdl	bdl	bdl
S-SO/ ⁻	mg/⊑	40 5	37.0	37.5	12.4	40 3	46.3
	mg/L	24.2	22.4	24.2	22.7	46.7	-10.0 20 1
	mg/L	24.Z	22.4 45.5	24.2	52.0	40.7	55.9
	mg/∟	40.0	40.0	44.5	0.0	49.7	0.0
wg	mg/L mg/l	12.0	12.3	12.4	9.0	0.1	9.3
Alkalinity	CaCO ₃	151.0	173.5	162.7	137.4	161.1	155.4
Anionic surfactants	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
Cationic surfactants	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
Non-ionic surfactants	mg/L	bdl	bdl	bdl	bdl	bdl	bdl
Hydrocarbons, oils and fats	mg/L	nd	bdl	nd	bdl	bdl	bdl
E. coli	CFU/100 mL	0.6	0.0	0.0	0.0	0.0	25.7
Legionella spp.	CFU/L	nd	0	nd	0	0	0

 Table 5.33. IP average efffluent quality for three HLs

bdl=below detection limit, nd=not determined

The IP effluent quality was excellent, presenting values that were bdl for almost all of the parameters. Therefore no effects could be seen for the HL in terms of quality of the effluent for the majority of the parameters (organic mater, solids, nutrients, ions, cations, detergents, hydrocarbons, oil and fat, *E. coli, Legionella* spp.). These results suggest that the DF+IP combination can provide high quality effluents up to HL of 36 cm/day. The effect of the fractionation of the HL was not observed for almost all the parameters. Only DO and turbidity presented differences. When increasing fractionation for the same HL, DO was slightly lower as was the turbidity. These results may be explained (as commented in the section on IP hydraulics) due to the greater fractionation with the same daily flow causes water to percolate more slowly and

therefore the oxygenation is lower and the filtration capacity is greater. These results coincide with studies of VFCW (Torrens *et al.*, 2009a).

5.4.2.5. Characterisation and effects of deposits from carwash effluents on the pilot plant

After one year in operation, deposits were formed in different parts of the pilot system (valves, HFCW inlet area and emitters from the IP system (Figure 5.22).



Figure 5.22. Deposit accumulation in the inlet (HFCW) and in the dripper emitters (IP)

These "biosolid" accumulations (of black colour) were characterised (Table 5.34 and 5.35). These deposits accumulated slowly in the HFCW inlet zone, clogging the system after one year in operation (water flooding in the inlet). After sampling, it was removed, and the inlet gravel was cleaned. In the case of IP these "biosolids" caused problems in the drippers, and the system was changed to other type of drippers with a purging system (as explained in section 5.3.3.1). Since its installation (and periodic purges, weekly or every two weeks), there has been no clogging. There has not been any clogging in the VFCWs. Part of influent solids is retained on the filter surface and creates a thin layer, as it is common in these systems. Moreover, distribution of the VFCW water makes it less likely to clog, as the water is distributed evenly all over the surface of the filter. This thin layer (less than 2 cm after one year in operation) has been formed in some parts of the HSFCW's surface, without causing any hydraulic problem.

Parameter	Value
DM	17.1 %
TOC	9.9 %
VS	17.9 %DM
Oils and fats	0.08 %DM
рН	5.9

 Table 5.34.
 Deposit characterisation

	Table 5.35.	Deposit	characterisation	(metals)
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Metal	Value (mg/Kg DM)
Antimony	41
Arsenic	<10
Cadmium	<2.0
Copper	3623
Tin	68
Mercury	<0.20
Nickel	57
Lead	114
Selenium	<10
Thallium	<4
Tellurium	4
Zinc	1642

The results of the deposit characterisation show VS percentages lower than the percentages found in sludge, algae deposits or swine slurry deposits (sections 3.4.1.3.4. and 4.4.2.6). The percentage of VS in swine slurry deposits or algae deposits ranged from 40 to 60% of VS. The percentage of VS of the deposits in the car washing pilots was only 17.9%. The percentage of total carbon is also low (9.9% TOC). Therefore, the percentage of organic matter was not high. DM values were low (17.1%) representing a material with high water content. The percentage of oils and fats is of 0.081%. As discussed in section 5.4.1, most of oils and fats were not pumped to the pilots (floated and passed on to the second ST). The high concentration of metals (particularly copper and zinc) is also remarkable. Metal monitoring will be carried out in the second stage of the project. There are numerous experiences with CWs (e.g. Gillepsie *et al.*, 2000) demonstrating their ability to eliminate them by using macrophytes (phytoremediation).

The HFCW clogging occurred with low organic and SS surface loading rates values. SS surface loading rates were on average of 2.9 gSS/m²·day with a maximum 8.3 gSS/m²·day. COD loading rates were on average of 3.5 gCOD/m²·day with a maximum 10.1 gCOD/m²·day. HFCWs likelihood of clogging is well known; however, this technology usually receives much higher SS and organic loadings than the studied pilot without clogging problems. The fact that the inlet distribution of the modular HFCW is only with one pipe makes this system more sensitive to clogging in the distribution area. However, similar HFCWs (with the same inlet design) treating domestic wastewater did not have any clogging problems with higher organic and SS surface loading rates (Torrens, 2013b). This means that fat and oils content (even at low concentrations) quickly clog HFCWs inlet areas or drip irrigation systems. As a result, it is advisable to remove fats and oils before application of these technologies.

5.5. Conclusions

The viability of SSFCWs to treat car wash effluents has been examined. The performance of the IP system was also evaluated in order to compare the removal efficiencies of both technologies.

The car wash influent (feeding the pilots) had the typical characteristics of water from car wash facilities, with slightly lower values for almost all of parameters: especially inorganic SS, fats and oils. The pilot plant inlet values correspond to wastewater that was impelled from a pump located in a settling tank and therefore settling has occurred (mainly sand and fines). SS in the influent of the pilots was approximately 41 mg/L (with 50% of VS). Wastewater at the inlet of the settling tank presented SS values > 300 mg/L with 10% VS.

The hydrocarbons and fats passed on considerably to the second settling tank and therefore did not reach in full to the pilots. Hydrocarbons, oils and fats concentrations from samples taken before the settling tank were roughly 1-2 mg/L. However, in the pilot's influent they were 0.2 mg/L, on average. The biodegradability of the influent was somewhat low (BOD₅/COD=0.3), with values similar to those of urban runoff waters. The influent presented variable COD contents ranging from (10-158 mg/L) and BOD₅ (5-50 mg/L).

E. coli concentrations were quite variable, ranging from 0 to 4.3 Ulog. The nutrient concentrations were very low (averages of 4.2 mg/L, 0.3 and 0.4 mg/L for TKN,
ammonia and phosphates respectively). The influent water presented only non-ionic surfactants but at lower concentrations than anticipated (maximum of 0.6 mg/L, average of approximately 0.2 mg/L). This was due to the high dilution, low dosing of detergents and high biodegradability of the used detergents.

The three pilot plants (HFCW, VFCW and DF+IP) performed very efficiently during the first year of the study for all of the applied loads (the maximum HL applied to the HFCW of 14 cm/day, and 36 cm/day for VFCW and IP). The three technologies had very high removal performance for turbidity, organic matter (COD, CODd and BOD₅) and solids (SS and VS). For these parameters, the pilot plant having the best removal efficiency was the DF+IP system (effluent values almost always below the technique's detection limit). Removal percentages for the organic matter parameters were also quite high for the VFCWs with an average of 78% for total COD (slightly greater than those of the HFCWs having an average value of 73%) even with the much larger hydraulic loads applied to the VFCWS.

DF+IP combination resulted in the almost complete elimination of SS and turbidity (bdl for SS) and average of 1.1 FNU for turbidity. The second system in terms of treatment performance for these parameters was the HFCW (with average effluent values of 4.7 mg/L for SS and 2.8 FNU for turbidity). Finally, the slightly lower degree of removal for VFCWs in this pilot plant is most likely due to the larger particle size of the sand (as compared to the IP) and the feeding mode. The VFCW had an average effluent of 11.7 mg/L for SS and 16.2 FNU for turbidity. It was observed that over the same batch, the effluent presented some variability in regards to turbidity depending on the time of sampling.

Nutrient concentrations were quite low in the influent, thus *Phragmites australis* growth in the CWs pilot plants was much slower than in other types of wastewaters (especially in the HFCWs in the areas farthest from the residual water inlet). The VFCW and IP nitrified the effluents. The HFCW, on the other hand, presented more anaerobic conditions and therefore did not produce nitrates. Nitrogen and phosphates were almost completely eliminated in the three pilot plants. The nutrient concentrations were very low, usually bld. No major variations were found in any of the pilot plants for the parameters of calcium, magnesium, sulphates, alkalinity and for electrical conductivity.

Surfactants present in the inlet of the pilot plants (non-ionic detergents) were 100% removed in the three pilots, but it should be noted that the concentrations of these

detergents were quite low in the influent. The same occurred with the oil, fats and hydrocarbons: they were efficiently eliminated by the three pilot plants, but their influent concentrations were very low, since the majority of these components pass through a second decanter and were not passed on to the pilot plants.

Regarding microbiological parameters presented average reductions of 1.4, 2.1 and 2.4 Ulog of *E. coli* corresponding to the HFCW, VFCW and DF+IP respectively. The average effluent values of *E. coli* in the three pilot plants were lower than those in the Royal Decree guidelines for reuse in Spain (200 CFU/100mL). The outlet values of the VFCWs and IP in all samples were lower than 200 CFU/100mL. For the HFCWs, the values exceeded 200 CFU/100mL in only 12 % of the samples. *Legionella* spp. was always absent in the inlet and outlet of the pilot plants except in the case in the effluent of the VFCW.

The VFCW pilot plant performed without any clogging problems throughout the study for all of the applied loads even without the application of resting periods. During the first year of operation, there was no sign of clogging of the filter matrix in the VFCW and IP, even with the highest HLs. However, for the IP, the irrigation system clogged after 10 months of operation with HL of 36 cm/day, despite good disk filter operation and low SS and COD values. This clogging was caused by biosolid accumulation.

The clogging deposit presented a DM content of approximately 17%, TOC 10% and VS 18% (of DM). The oils and fats percentage was 0.081% DM. There were also notably high concentrations of metals (particularly copper and zinc). These deposits accumulated slowly in the HFCW inlet zone causing system clogging after 1 year of operation (water flooding in the inlet). Clogging in the HFCWS occurred at very low loading rates (average 2.9 gSS/m²·day, maximum of 8.3 gSS/m²·day, and 3.5 gCOD/m²·day, maximum 10.1 gCOD/m²·day). Regardless of the low oils and fats content reaching the pilots, these components had a high clogging capacity for the HFCWs, valves and dripper emitters of the IP system. Therefore the oil and fats contents of the car wash effluents as well as the inorganic suspended solids made pretreatment necessary.

The water balances carried out have revealed losses via evapotranspiration of 8 to 12 % for the HFCWs and practically zero losses for the VFCWS and IP. This suggests that almost all of the inlet wastewater may potentially be recycled in the car wash.

The effect of the HL on effluent quality was examined for each pilot plant. Upon increasing hydraulic load, the most sensitive technologies were found to be the HFCW for the organic mateter parameters and the VFCWS for SS and turbidity. An increased HL decreases removal efficiency. There was no significant effect of hydraulic load on IP effluent quality (with values bdl for almost all the parameters).

The fractionation of the hydraulic load did not affect average purification performances in the VFCWs, but a greater fractionation resulted in more regular effluent quality, mainly for SS and turbidity. For the IP, the DO and turbidity parameters were only slightly affected. In the IP, upon increasing the fragmentation for a single hydraulic load, a slight decrease was observed in DO and turbidity of the effluent.

The study has revealed the viability of SSFCWs for the treatment of car wash wastewater previously settled and for which oils and fats were previously eliminated, and to produce a very high quality effluent that may be recycled within the system (in processes requiring less exigent water quality and that have greater water consumption: prewash with hand-held lances and first wash step with brush arches). SSFCWs have been found to adapt to the pollutant and HL fluctuations (mainly VFCWs). However, a final chlorination step is recommended in order to ensure the presence of disinfectant in the pipes that transports the water to the car wash facility.

More detailed studies should be carried out in regards to the effect of certain potentially corrosive pollutants (metals, salts) on the machinery used in the facilities.

6. CONCLUSIONS

6. CONCLUSIONS

The **main conclusion** of the thesis is the viability of the application of different configurations of subsurface flow constructed wetlands to treat the effluents from a wastewater treatment pond, a pig farm, and a car wash facility, once design and operation have been optimized.

In **general**, subsurface flow constructed wetlands have proved to be a sustainable and efficient technical solution to treat small wastewater flows with special characteristics. Subsurface flow constructed wetlands have shown resilience to load and hydraulic fluctuations, to new pollutants and to environmental variable conditions; being simple to operate and maintain with null or minimum energy requirements and with an added aesthetical value.

Specific conclusions have been drawn from each of the three parts of the study.

- Wastewater characterisation is a key factor for the design of subsurface flow constructed wetlands. Pond effluents present high variability and large quantities of algae, dissolved and particulate organic matter with good biodegradability, and suspended solids. Swine slurry contains high concentrations of suspended solids, organic matter, nitrogen (mainly ammonia), phosphorous and *E. coli*. Finally car wash effluent contains high concentrations of inorganic solids, very variable concentrations of *E. coli* and organic matter, hydrocarbons, fats, oils and low concentrations of nutrients and non-ionic surfactants.
- The studies have proved the effectiveness of vertical and horizontal flow constructed wetlands upgrading the pond effluent quality by retaining algae and suspended solids, completing organic matter degradation, and nitrifying the pond effluent in the case of vertical filters or partially removing total nitrogen in the case of horizontal filters. Retention of phosphorus was low and decreased with time. Removal of algae in both vertical and horizontal flow constructed wetlands depends on the suspended solids particle size, and thus on the algae genera. The granulometry laser technique to determine the size and number of particles in the water samples is a useful method to characterise wastewater suspended solids and to study the filtration capacity on subsurface flow constructed wetlands.

- Removal of microbiological indicators in vertical subsurface flow constructed wetlands ranged from 0.5-1 Ulog for viral indicators to 1-2 Ulog for bacterial indicators. Bacterial indicators are removed at a higher rate than viral ones. Somatic coliphages are removed at higher rates than F-specific bacteriophages. Low temperatures do not limit the removal of indicator microorganisms in vertical flow constructed wetlands.
- Monitoring of infiltration rates is a useful tool to study the filters' hydraulic performance. Infiltration rates decrease in vertical flow constructed progressively with every successive batch (feeding). Tracer tests allow determining the mean hydraulic retention times and the detention time distribution curves in the vertical flow constructed wetlands. The mean hydraulic retention times varies depending on the filter's design and operation.
- The hybrid system (vertical + horizontal flow constructed wetland) treating swine slurry presents a dual function (solid-liquid separation and biological treatment) and has achieved a significant removal of nitrogen, organic matter, suspended solids and partial disinfection. The vertical flow constructed wetlands, operated intermittently and with sequential feeding, demonstrated good hydraulic performance without clogging problems, despite high pollutant loads.
- The capacity of nitrification/denitrification of a hybrid constructed wetland treating swine slurry has been proved: the overall nitrogen reduction was 63 % on average. The vertical flow constructed wetland has demonstrated their effectiveness nitrifying the swine slurry, removing approximately 42% of TKN and producing high nitrate concentrations. The average removal rates for the horizontal flow constructed wetland were approximately 40% for TKN and 65% for NO₃⁻. The correlation between nitrification and temperature resulted in a higher percentage of elimination when temperatures increased. The percentage of nitrogen removal in the study allows increasing the volume of effluent applied in agricultural land, improving sustainable swine slurry management at the farm.
- The three technologies treating car wash effluent (horizontal flow constructed wetland, vertical flow constructed wetland and the infiltration-percolation) demonstrated their effectiveness with high percentages of removal of turbidity, organic matter and suspended solids. Infiltration-percolation system has the best performances regarding the above mentioned parameters with effluent values almost always below detection limit. The three pilots (particularly the infiltration-percolation).

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percolation and the vertical flow constructed wetland) remove *E. coli* and reach acceptable limits for water reuse, with average effluent concentration lower than 200CFU/100 mL.

- The studied car wash effluent contained only non-ionic surfactants at low concentrations that were removed in the subsurface flow constructed wetlands and the infiltration-percolation system. The small amount of hydrocarbons, oils and fats present in the inlet of the pilot plants were eliminated below detection limit by the three technologies. Hence, the removal of surfactants and hydrocarbons could not be studied in detail due to their low influent concentrations. No significant variations were found in any of the pilot plants for calcium, magnesium, sulphates, alkalinity and for electrical conductivity.
- Subsurface flow constructed wetlands and the infiltration-percolation treating car wash effluents produce a very high quality water that can be recycled within the system (in processes requiring less demanding water quality and that have greater water consumption: prewash with hand-held lances and first wash step with brush arches). A final disinfection (i.e. chlorination) is recommended in order to ensure the presence of (residual) disinfectant in the recycling pipes. More detailed studies are recommended to investigate the effect of recycling water that contains corrosive pollutants (e.g. metals, salts) to the machinery in the facilities.
- Differences in performances of subsurface flow constructed wetlands were evident, based on the design and operation. The influence of design parameters (depth, media type and size, presence of plants) as well as operational parameters (hydraulic load, feeding regime) on filter performance was determined.
 - The filtering media size (sand or gravel) is the key parameter for the algae retention.
 - The choice of the sand (d₁₀ and coefficient of uniformity) is especially important for vertical flow constructed wetlands, in order to obtain a good filtration, provide enough retention time and avoid clogging.
 - Crushed sand filters performed worse (lower algal retention and percentage removals for physicochemical parameters) than river sand filters in all the tested conditions.

- The presence of plants does not significantly affect the filter performances (in terms of both physicochemical and microbiological parameters), although it is important for temperature moderation.
- For vertical flow constructed wetlands, the deeper the filters the better the performance for all physicochemical and microbiological parameters, and especially for the removal of organic matter and for algae retention.
- An increase in hydraulic load has led to reduce removal efficiency of the majority of parameters in both types of subsurface flow constructed wetlands.
- Fractionation of the daily hydraulic load influenced hydraulic retention time and infiltration rates in vertical flow constructed wetlands and was proved to have an important role in determining the treatment level.
- The removal of bacterial and viral indicators in vertical flow constructed wetlands depends mainly on the water retention time in the filter, which in turn depends on the media granulometry, on the depth of the filter and on the hydraulic load and the dose volume per batch.
- The control of the surface deposits layer by controlling the feeding and resting periods as well as the maximum hydraulic load and surface loading rates are of great importance for the durability and the reliability of vertical flow constructed wetlands.
 - In pond effluents, failure to respect the recommended feeding and resting periods can lead to formation of clogging deposits on the surface of the vertical flow constructed wetlands. These organic deposits composed by algae, strongly decrease infiltration rates and hinder oxygenation resulting in a decrease of performance in terms of nitrogen and dissolved chemical oxygen demand and an increase of suspended solid removal and algae retention.
 - In the vertical flow constructed wetland treating swine slurry, some of the contaminants were retained, dried and mineralized in the surface of the vertical flow constructed wetland. This biosolid organic layer, that increased around 20 cm per year, improved filtration efficiency, and thus solids removal, water retention time in the system and treatment performances. This deposit presents high concentrations of organic solids and nutrients that could be reused as a sub product for compost or fertilizer.

- The drawbacks of subsurface flow constructed wetlands for each type of wastewater have been determined.
 - High ammonia contents in swine slurry (applied loads to the vertical flow constructed wetland of 105 g NH₄⁺/m²·day) together with high organic loads represent a major limitation for the *Phragmites australis* development. On the other hand, low concentrations of nutrients in car wash wastewater results in a slow growth of *Phragmites australis*.
 - The high concentrations of suspended solids and organic matter limits the type of subsurface flow constructed wetlands to be implemented. Vertical flow constructed wetlands operated intermittently with resting periods is the most convenient option to treat high strength effluents.
 - The studies have proved the need to remove fat and oils and the inorganic solids prior to the application of subsurface flow constructed wetland in order to avoid media clogging. The horizontal flow constructed wetland treating car wash effluents demonstrated to be more sensitive to the clogging.

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APPENDIX A: ANALYTICAL METHODS

Next tables show water quality and deposits/sludge characterisation parameters and the analytical methods used in each study site.

 Table A1. Water control parameters and analytical reference methods (study site 1: Aurignac WWTP)

Parameter	Method
рН	4500-H+B (Standard Methods, 2005)
EC	2510-A (Standard Methods, 2005)
Temperature	2550-A (Standard Methods, 2005)
COD	NF T 90-101 (AFNOR, 2005)
BOD ₅	NF T 1899-1 (AFNOR, 2005)
SS	NF EN 872 (AFNOR, 2005)
N-NH4 ⁺	NF T 90 015 2 (AFNOR, 2005) - when measurements were taken on site the ammonium was analysed with a spectrophotometer WTW Photolab S12 following the Nessler method.
TKN	NF EN 25-663 (AFNOR, 2005)
N-NO3 ⁻	NF EN 10-304 (AFNOR, 2005) - when the measurements were taken on site the nitrates were analysed with a spectrophotometer WTW Photolab S12. KIT WTW 14542
TP	NF EN 1189 (AFNOR, 2005)
P-PO4 ³⁻	NF EN 1189 (AFNOR, 2005)
Particle count and size distribution	Light-scattering method. Laser beam as source of light. Particle-counter and size distribution analyzer HYAC/ROYCO 8000 A (Pacific Scientific) - particle size limits: 1-150 μm.
E. coli	Filtration method ISO 9308-1 (ISO, 2000)
Fecal coliforms	Filtration method 9222-D 2510B, (Standard Methods, 2005)
Somatic coliphages	ISO 10705-2 (ISO, 2000) E. coli CN ATCC 700078 was used as the host strain
F-specific bacteriophages	ISO/CD 10705-1 (ISO, 1995), <i>Salmonella typhimurium</i> NCTC 12484 was used as the host strain

Parameter	Method
Chlorophyll-a	Determined according to the spectophotometric method for the particulate fraction of the samples, after filtration (0.45-µm-pore-size filter) and extraction with acetone 90% method NT 90-117 (AFNOR, 2005).
Algal identification	Whenever possible, the microscopic examination was perforemd the day of collection. If this was not possible, samples were conserved in formaldehyde 4% at 4°C. Algae genera were identified using a Zeiss Axioplan microscope, with a magnification of x100 to identify the large cells and a magnification of x400 for the smaller algae. The dominant genera of algae were identified as far as possible referring to Bellinger (1980). Images of the samples were taken using the MRc5 AxioCam photomicrographic system.

Table A2. Algal biomass monitoring in water samples (study site 1: Aurignac WWTP)

 Table A3. "Algal deposit" control parameters and analytical reference methods (study site 1, Aurignac

Parameter	Method
DM	Oven Drying at 105 °C
VS	Ignition at 550 °C

Table A4. Water control parameters	and analytical reference methods (study site 2: Santa
	Eugènia WWTP)

Parameter	Method
рН	4500-H+B (Standard Methods, 2005)
EC	2510-A (Standard Methods, 2005)
Temperature	2550-A (Standard Methods, 2005)
COD	Adaptation of 410 54 US EPA method (Hanna, www.hannainst.es)
SS	2540-D (Standard Methods, 2005)
$N-NH_4^+$	D1425-92 (Hanna, www.hannainst.es)
TN	D1425-92 (Hanna, www.hannainst.es)
N-NO ₃ ⁻	Cromotropic acid method (Hanna, www.hannainst.es)
TP	Adaptation of 4500-C (Standard Methods, 2005)
Particle count and size distribution	Light-scattering method. Laser beam as source of light. Particle-counter and size distribution analyzer HYAC/ROYCO 8000 A (Pacific Scientific). Particle size limits: 1-150 µm.
E. coli	Filtration method ISO 9308-1 (ISO, 2000)

Parameter	Method
Chlorophyll-a	Determined according to the spectophotometric method for the particulate fraction of the samples, after filtration (0.45-µm-pore-size filter) and extraction with acetone 90 method NT 90-117 (AFNOR, 2005).
Algal identification	Whenever possible, the microscopic examination was perforemd the day of collection. If this was not possible, samples were conserved in formaldehyde 4% at 4°C. Algae genera were identified using a Zeiss Axioplan microscope, with a magnification of x100 to identify the large cells and a magnification of x400 for the smaller algae. The dominant genera of algae were identified as far as possible referring to Bellinger (1980). Images of the samples were taken using the MRc5 AxioCam photomicrographic system.

Table A5. Algal biomass monitoring in water samples (Study site 2: Santa Eugènia WWTP)

Table A6. Water control parameters and analytical reference methods (Study site 3: "Car
Coromines" pig farm)

Parameter	Method
Temperature	2550-A (Standard Methods, 2005).
рН	4500-H+B (Standard Methods 2005)
EC	2510-B (Standard Methods, 2005)
COD	5220-C (Standard Methods, 2005)
BOD₅	5210-B (Standard Methods, 2005)
SS	2540 D (Standard Methods, 2005)
TKN	4500-Norg (Standard Methods, 2005)
N-NH4 ⁺	4500-NH ₃ (Standard Methods, 2005)
N-NO ₃ ⁻	4500-NO ₃ (Standard Methods, 2005)
P-PO4 ³⁻	4500-P (Standard Methods, 2005)
E. coli	Filtration Method ISO 9308-1 (ISO, 2000)

Table A7. Sludge control parameters and analytical reference methods (Study site 3: "Can Coromines" pig farm)

Parameter	Method
DM	Oven drying at 105 °C
VS	Ignition at 550 °C
TKN	Distillation (MAPA, 1994)
P ₂ O ₅	Volumetric (MAPA, 1994)

Parameter	Reference method*
Temperature	Multiparameter Portable Meters (Hanna, www.hannainst.es)
pН	Multiparameter Portable Meters (Hanna, www.hannainst.es)
EC	Multiparameter Portable Meters (Hanna, www.hannainst.es)
Redox	Multiparameter Portable Meters (Hanna, www.hannainst.es)
Turbidity	Multiparameter Portable Meters (Hanna, www.hannainst.es)
COD	Spectrophotometry Ca-R-PE-0002 (LABAQUA SA, www.labaqua.com)
BOD₅	Manometric Ca-R-PE-0001 (LABAQUA SA, www.labaqua.com)
SS	Gravimetric Ca-R-PE-0005 (LABAQUA SA, www.labaqua.com)
VSS	Gravimetric Ca-R-PE-0005 (LABAQUA SA, www.labaqua.com)
TKN	Distillation and titration Ca-R-PE-0008 (LABAQUA SA, www.labaqua.com)
N-NH4 ⁺	Volumetric A-F-PE-0019 (LABAQUA SA, www.labaqua.com)
N-NO ₃ ⁻	Electrode Ca-R-PE-0001 (LABAQUA SA, www.labaqua.com)
P-PO4 ³⁻	Chromatography Ca-C-PE-0001 (LABAQUA SA, www.labaqua.com)
SO4 ²⁻	Chromatography Ca-C-PE-0001 (LABAQUA SA, www.labaqua.com)
Cl	Chromatography Ca-C-PE-001 (LABAQUA SA, www.labaqua.com)
Ca ²⁺	ICP Ca-C-PE-002 (LABAQUA SA, www.labaqua.com)
Mg ²⁺	ICP Ca-C-PE-002 (LABAQUA SA, www.labaqua.com)
Alkalinity	Volumetric Ca-Q-PE-0029 (LABAQUA SA, www.labaqua.com)
Anionic surfactants	Spectrophotometry Mad-G-PE-0223 (LABAQUA SA, www.labaqua.com)
Cationic surfactants	Spectrophotometry Mad-G-PE-0226 (LABAQUA SA, www.labaqua.com)
Non-ionic surfactants	Spectrophotometry Mad-G-PE-0227 (LABAQUA SA, www.labaqua.com)
Hydrocarbons, oils and fats	IR A-F-PE-005 FTIR (LABAQUA SA, www.labaqua.com)
E. coli	Filtration Ca-M-PE-0046 (LABAQUA SA, www.labaqua.com)
Legionella spp.	Filtration Ca-M-PE-0059 (ISO 11731) (LABAQUA SA, www.labaqua.com)
Intestinal nematoda	Sample concentration and microscopic observation (A-E-PE-0034)
	(LABAQUA SA, www.labaqua.com)

 Table A8. Water control parameters and analytical reference methods (Study site 4: "Montfullà" car wash)

Parameter	Reference method
DM	Gravimetric A-F-PE-0013 (LABAQUA SA, www.labaqua.com)
TOC	Gravimetric A-F-PE-0068 (LABAQUA SA, www.labaqua.com)
VS	Gravimetric A-F-PE-0068 (LABAQUA SA, www.labaqua.com)
Oils and fats	IR A-F-PE-0005 (LABAQUA SA, www.labaqua.com)
Hydrocarbons	IR A-F-PE-005 (LABAQUA SA, www.labaqua.com)
Antimony	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)
Arsenic	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)
Cadmium	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)
Copper	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)
Tin	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)
Mercury	AFS A-D-PE-0005 (LABAQUA SA, www.labaqua.com)
Nickel	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)
Lead	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)
Selenium	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)
Thallium	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)
Tellurium	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)
Zinc	ICP A-D-PE-0025 (LABAQUA SA, www.labaqua.com)

Table A9. "Deposit" control parameters and analytical reference methods (Study site 3:"Montfullà" car wash)

IR: Infrared Spectroscopy, ICP: Inductively Coupled Plasma, AFS: Atomic Fluorescence Spectroscopy

APPENDIX B: TRACER TESTS

- Tracer

The tracer used for the tests was sodium chloride (NaCl). The choice of tracer is the first thing to define when preparing a tracing experiment. For practical and financial reasons, the most commonly used tracing methods are conductimetric methods. The salt most commonly employed for this type of tracing is sodium chloride (NaCl) because of its comparatively low cost and its properties (is not toxic and therefore does not harm the environment). The principal limitation in the use of this tracer is the possibility of salt retention by the biomass of the medium. However, with the amount of salt used, 1.5 kg per 1 m³ of batch volume, the highest tracer concentration present is relatively low (1.5 g/L) and under these conditions the influence on the attached biomass is considered to be negligible.

- Test methodology

In previous studies (Molle, 2003), the use a 1-minute time interval was concluded to be a good choice between the precision of the measurements and the quantity of data produced. Therefore we measured the conductivity and flows at the outlet of the filters every minute to monitor the tracer. Each tracer test (except tests 9 and 10) was carried out simultaneously on the two filters containing the same medium but with different heights due to the feed operation of the filters. For the calculations each bed was assumed to receive half the volume of each batch. The outlet flows of the two filters studied in each test were measured separately. One filter flow was measured with the station flowmeter and the other with a portable flowmeter.

The experiments were performed after fully moistening the filter (4 batches had to precede the tracing batch) and for the entire duration of the batch. After the tracer was injected, batches without tracer were delivered to the filter (following the normal operating schedule) until the salt was totally recovered at the outlet.

The tracer batch was prepared in the tank that feeds the filters. A conductivity meter was installed in the feed post, and salt was added until the conductivity reached 2 ds/m. A pump was also installed to mix the salt throughout the batch. The tracing batch was then applied as a pulse in one hydraulic loading interval (batch) to the filters. After
this, the feed tank was washed with tap water in order not to contaminate successive batches with salt. At the outlet of each filter, the flow and the conductivity were continuously measured and recorded until almost all the salt was recovered.

- Tracer test parameters

Several parameters were calculated for ech tracer test. This allows to compare the behaviour of the filters under different conditions.

- Detention time distribution

The residence time distribution curve (RTD) is not applicable as operation is not under a permanent regime. However, it is possible to determine the detention time distribution curve (DTD), ψ (t)=f(t), defined as follows:

$$\psi(t) = \frac{Q(t)}{M} \cdot C(t)$$

Where:

Q (t) is the outlet flow (L/sec);

C (t) is the average concentration of the tracer at the outlet at a given time(g/L); and M is the mass of tracer injected (g).

This method does not let us describe the hydraulics of the system with the classical models (plug-flow reactor, perfectly mixed flow reactor, etc.) because of this non-continuous feed regime. However, the DTD allows us to observe the intensity of the exchanges, the heterogeneity of the flow and the detention time of the tracer in the reactors.

- Mean residence time

From the DTD, the mean residence time (t) can be estimated. The mean residence time is the average of the detention times of every particle in the filtering media, and is defined by the following equation:

$$\bar{\mathbf{t}} = \frac{\int_{t=0}^{\infty} t \cdot \psi(t) \cdot dt}{\int_{t=0}^{\infty} \psi(t) \cdot dt} \quad ; \quad \int_{t=0}^{\infty} \psi(t) \cdot dt = 1 \text{ by definition, hence } \bar{t} = \sum_{i=0}^{\infty} t_i \cdot \psi(t_i)$$

- Minimum retention time

The minimum retention time was defined as the time taken to reach 10% of initial salt concentration in the filter effluent. The use of a minimum retention time instead of the mean retention time is reported to be more appropriate for predicting the removal of microorganisms based on breakthrough curves in unsaturated filters. Therefore, this parameter was also calculated to estimate the relationship with the removal of bacterial indicators.

- Dilution of the tracing batch

The mass of salt recovered at the outlet of the filters (by means of its concentration and the volume of the recovered liquid) allows us to determine (for each batch) the volume of water coming from the tracing batch and the volume of water which does not come from the tracing batch, but from the water stored in the filter. This is possible id we suppose that the concentration of the tracer measured at the outlet of the filter depends on the concentration of the tracer at the inlet and on a dilution factor induced by the mixing that occurs in the porous media. Hence we can define the following relations:

$$V(t) = V_{coming from the batch} + V_{coming from the filter}(t)$$

$$C(t) = \frac{V_{coming from the batch} \cdot C_{tracing batch}}{V(t)}$$

Where:

V (t) is the volume of water at the outlet at a given time t (L);

 $V_{coming from the batch}$ is the volume of water from the tracing batch (L);

 $V_{not \ coming \ from \ the \ batc^{\circ}h}$ is the volume of water stored in the filter (L);

C (t) is the concentration of the tracer at the outlet at a given time t (g/L); and

 $C_{tracing batch}$ is the concentration of the tracer in the tracing batch (g/L).

Consequently,

$$V_{coming from the bacth}(t) = \frac{C(t) \cdot V(t)}{C_{tracing bacth}}$$

This calculation makes it possible to quantify the dilution of the tracing batch within the filtering media. The monitoring of this parameter over time allows us to determine the heterogeneity of the water flow (Molle, 2003).

APPENDIX C: SAND GRANULOMETRY CURVES

Next figures show the sand granulometry curves (particle size distribution curves) for each study site.



Figure C.1. Particle size distribution curves (crushed and river sand; Aurignac WWTP study site)



Figure C.2. Particle size distribution curve (VFCW sand; Santa Eugènia WWTP study site)



Figure C.3. Particle size distribution curve (VFCW sand; Montfullà car wash facility study site)



Figure C.4. Particle size distribution curve (IP sand; Montfullà car wash facility study site)

LIST OF PUBLICATIONS

- Peer-reviewed journals

- Published

Torrens, A., Molle, P., Boutin, C. and Salgot, M. (2009). Removal of bacterial and viral indicators in vertical flow constructed wetlands and intermittent sand filters. *Desalination*, 246: 169-178.

Torrens, A., Molle, P., Boutin, C. and Salgot, M. (2009). Impact of design and operation variables on the performance of vertical-flow constructed wetlands and intermittent sand filters treating pond effluent. *Water Research*, 43: 1851-1858.

Turon, C., Comas, J., Torrens, A., Molle, P. and Poch, M. (2008). Improvement of sand filter and constructed wetland design using an environmental decision support system. *Journal of Environmental Quality*, 37(4): 1644-1647.

- Submitted

Torrens, A. Bayona, C. Salgot, M. and Folch, M. Performance, design and operation of hybrid subsurface flow constructed wetland for swine slurry treatment. Submitted to *Ecological engineering*.

Torrens, A., Folch, M., Salgot, M. and Aulinas, M. Comparison of natural technologies for the treatment and reclamation of car wash facilities effluents. Submitted to *Ecological engineering*.

Congress and conferences contributions

Folch, M., Torrens, A., M., Salgot, Agulló, N., Borrós, S. and Aulinas, M. (2015). Projecte de demostració d'estalvi d'aigua en el rentat de cotxes mitjançant l'ús de detergents innovadors i el tractament natural d'aigües residuals. I Congrés de l'Aigua a Catalunya, 18-19 March 2015, Barcelona, Spain.

Torrens, A., Folch, M., Bayona, C. and Salgot, M. (2013). Upgrading quality of wastewater by means of decentralized natural treatment systems. Sustainable management of environmental issues related to water stress in Mediterranean islands. Mediwat Project. 24th May 2013, Palermo, Italy. Oral.

Gallinas, M., García, M., Torrens, A. and Salgot, M. (2013). Upgrading the effluent of storage ponds to achieve quality standards for irrigation purposes. Sustainable management of environmental issues related to water stress in Mediterranean islands. Mediwat Project. 24th May 2013, Palermo, Italy. Oral.

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Salgot, M. and Torrens, A. (2012). Natural and simplified systems for wastewater treatment and reuse and their adaptation at small sites. Sustainable management of environmental issues related to water stress in Mediterranean islands. Mediwat workshop, 15th March 2012, Aix de Provence, France. Oral.

Torrens, A., Folch, M., Lucero, M. Sasa, J. and Salgot, M. (2012). Natural technologies combination for wastewater treatment and reuse in Catalonia. International Conference on advanced wastewater treatment technologies and the potential of phytoremediation technology for wastewater treatment in Vietnam. Hanoi, Vietnam, 2013. Oral.

Torrens, A. Folch. M. and Salgot, M. (2011). Constructed wetlands for wastewater treatment and reuse in Catalonia. 1st Phytoremediation international Congres, 4th November 2011, Hanoi, Vietnam. Oral.

Lucero, M., Sasa, J., Torrens, A., Folch, M., Huertas, E. and Salgot, M. (2010). Natural systems for wastewater treatment and reuse in small communities in Catalonia. 9th IWA Conference Sustainable solutions for small water and wastewater treatment systems (S2Small2010), 19-22 April, Girona, Spain. Oral.

Torrens, A., Folch, M., Sasa, J., Lucero, M., Huertas, E., Molle, P., Boutin, C. and Salgot, M. (2010). Removal of bacterial and viral indicators in horizontal and vertical subsurface flow constructed wetlands 12th IWA International Conference on Wetland Systems for Water Pollution Control, 3-8 October 2010, San Servolo, Venice, Italy. Oral.

Lucero, M., Sasa, J., Martins, P., Torrens, A., Folch, M., Tapias, J. and Salgot, M. (2009). Combination of non-conventional technologies for wastewater treatment and reuse in small communities. 3rd WETPOL Symposium on Dynamics and Control of Pollutants in Wetlands. 2009. Barcelona, Spain. Poster. Torrens, A., Molle, P., Boutin, C. and Salgot, M. (2007). Removal of bacterial and viral indicators in vertical flow constructed wetlands and intermittent sand filters. Multi functions of wetlands systems, 26-29 June, Padova, Italy. Oral.

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- Book chapters

Torrens, A., Folch, M., Bayona, C. and Salgot, M. (2013). Upgrading quality of wastewater by means of decentralized natural treatment systems Sustainable management of environmental issues related to water stress in Mediterranean islands. Noto, M. T., Vega, T. and López, A. eds. ISBN: 978-88-902822-3-2; pp. 136-144.

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Salgot, M. and Torrens, A. (2008). Tecnologías extensivas para la regeneración de aguas residuals. In: Aguas continentales. Gestión de recursos hídricos, tratamiento y calidad del agua. Damià Barceló (coordinator). Ed. CSIC, Spain, ISBN: 978-84-00-08664-0; pp. 197-225.