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Phosphorus and Nitrogen Removal by Saturated Vertical-Flow Constructed Wetlands (SVCWs) Type Micro-Pilot Using Shale from Ivory Coast as a Substrate

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Abstract

This study evaluates the performance of shale from Ivory Coast used as substrate in vertical-flow constructed wetlands in removal of phosphates and nitrogen. The pilot-scale artificial wetland has been duplicated: filter planted with *Panicum maximun* and unplanted. They were set up outdoors, and fed with a municipal wastewater. The wetlands have been fed with three batches per week (intermittent) over a period of 3 months. During the operation period, the hydraulic residence time (HRT) 52 h was used, while wastewater temperatures varied from about ~33°C. The removal performance of the constructed wetland units was very good, since it reached on an average 98%, 89.4%, 89.4%, 84%, 80%, 84.8% and 92% for TSS, DOC, BOD_5, NH_4^+, TKN, TP and $P\text{-}PO_4^{3-}$ respectively. In addition, the vegetation did not demonstrate superior performance to unplanted controls. Therefore, this study focuses on the role of shale in the phosphorus and nitrogen removal from wastewater by constructed wetland.

Keywords

Constructed Wetlands, Vertical Flow, Shale, *Panicum maximum*, Phosphorus Removal, Nitrogen

1. Introduction

Constructed Wetlands (CWs) are becoming increasingly popular around the world. This is for their performance, their hardiness, their ease of exploitation, and also their landscape integration (Vymazal, 2007; Ouattara et al., 2008; Zhao et al., 2011; Shama et al., 2015). However, this technology has been absent for a long time in Africa (WHO, 2003). Yet Africa faces enormous environmental problems, especially those related to municipal wastewater treatment. For good reason, the treatment systems (activated sludge, bacterial beds) installed since independence in 1960 have shown their inefficiency because of their complexity, their cost of operation and maintenance which cannot be ensured in the long term (Tuo et al., 2012; Dongo, 2001; Akpo et al., 2016). This implies the discharge of municipal wastewater into surface water without adequate treatment and is the basis for their degradation (e.g. eutrophication, disappearance of biodiversity, reservoirs of pathogenic micro-organisms) and makes them unfit for human consumption (Yao et al., 2009). In fact, eutrophication has been directly linked to the enrichment of aquatic environments with nutrients (mainly phosphorus and nitrogen). It manifests itself in the intensive development of algae and aquatic plants with the direct environmental consequence of a change in the balance of ecosystems, manifested by an impoverishment of biodiversity. The eutrophication phenomenon is accentuated by domestic or industrial agricultural discharges in aquatic environments. This is the case in Côte d'Ivoire where the Ebrié lagoon, a receptacle for all wastewater from Abidjan, is subject to advanced eutrophication. One of the immediate consequences is the degradation of the quality of this waterbody that has become unfit for human consumption (Yao et al., 2009). The treatment of nitrogen and phosphorus by Constructed Wetlands system to control eutrophication has been the subject of several studies (Drizo et al., 2000; Shama et al., 2015; Vymazal, 2007). Indeed, with respect of the removal of phosphorus (P), the results of several authors (Molle et al., 2011; Vohla et al., 2007; Vohla et al., 2011) showed in this system could be due to several phenomena: 1) adsorption on the filter mass, 2) bacterial action, 3) plant adsorption and incorporation into organic matter.

Major P removal mechanisms in CWs are adsorption and precipitation within the filter media (Babatunde et al., 2009). These processes largely depend on the physical-chemical properties of media materials (Vohla et al., 2011). Therefore, selection of wetland media with high P removal capacity is very important. Modified wetland medias containing calcium (Ca), iron (Fe), aluminum (Al) and magnesium (Mg) have attracted a lot of interest for enhancing P removal (Lan et al., 2018). For example, excellent P removal was observed in CWs with Al-based water treatment residual (Al-WTR) (Babatunde et al., 2009), magnesia (Lan et al., 2018), basic oxygen furnace (BOF) slag aggregates (Cai et al., 2012), thermally-modified calcium-rich attapulgite (Yang et al., 2006), maifanite and steel slags (Liu et al., 2011).

However, these authors agree that the adsorption on Fe, Al, Mn and Ca metal

rich substrates has a very large influence. Nitrogen removal is largely dependent on the supply of oxygen to the Constructed Wetlands system. The Constructed Wetlands system is continuously saturated with water and is therefore generally in anaerobic conditions. On the one hand, the plants carry oxygen to the rhizosphere, creating aerobic microsites adjacent to the roots and rhizomes in the otherwise reduced substrate. Ammonium ions can be oxidized by nitrifying bacteria (*Nitrosomonas*) to nitrite and then by *Nitrobacter* into nitrate ions. Conversely, at a greater distance from the roots, denitrification (conversion of nitrate to gaseous nitrogen) may occur in the anaerobic region (Brix, 1994; Drizo, 1997).

The aim of the present work is to evaluate the remediation of wastewater by using constructed wetlands (SVCWs) containing *Panicum maximum* and covered with shale from Ivory Coast under tropical climate.

2. Materials and Methods

2.1. Physico-Chemical Characteristics of the Substrate Material (Shale)

The shale used in this study was obtained from the Center region of Côte d'Ivoire (Toumodi. Lomo North) in order to evaluate its phosphorus removal in constructed wetlands. The particle-size distribution of the shale on a weight basis was analysed in triplicate by conventional dry-sieving technics. The grain-size distribution plots were used< to estimate d_{10} (10% of the sand by weight is smaller than d_{10}) and d_{60} (60% of the sand by weight is smaller than d_{60}). The uniformity of the particle-size distribution (the uniformity coefficient) was calculated as the ratio between d_{60} and d_{10}; according to the literature, materials of 0.2 - 1 mm effective size and uniformity coefficient less than 3 are appropriate for substrates of constructed wetlands (Knowles et al., 2011; Mallet et al., 2013). The corresponding values of shale presented in **Table 1** satisfy the above-mentioned requirements (Kpannieu et al., 2018). Porosities were determined from the amount of water needed to saturate a known volume of shale and the bulk density of the shale (g·cm^{-3}) were based on the ratio between the dry weight and the bulk volume of

Table 1. Physico-chemical characteristics of shale.

Chemical composition (%. w/w)										
SiO$_2$	Al$_2$O$_3$	Fe$_2$O$_3$	MnO	MgO	CaO	Na$_2$O	K$_2$O	TiO$_2$	P$_2$O$_5$	LoI**
56.8	17.5	10.2	0.6	2.1	0.4	0.9	2.4	1	0.1	8.7

Geometrical grading characteristics							
Shale (mm)	d_{10} (mm)	d_{60} (mm)	UC	Porosity (%)	Apparent density (g·cm^{-3})	Actual density (g·cm^{-3})	K* (m·s^{-1})
1-2	1.79	0.95	0.53	50	1.14	2.4	3.2×10^{-2}

Values for porosity, d_{10}, d_{60}, uniformity coefficient (UC) (d_{60}/d_{10}) and hydraulic conductivity (K*) are mean of triplicate analyses.

the sands. Saturated hydraulic conductivities were determined using the constant-head method in the laboratory. The Physico-chemical characteristics of shale is analyzed by (ICP-OES Varian 720-ES). The results of the analyzes are compared with the geological reference materials. The Loss on Ignition (PF) was determined by gravimetry on a wire mesh at 1000°C after passing through an oven at 110°C. The shale sample analyzed contains a significant amount of silicon oxide (56.8%). aluminum oxide (17.5%) and iron oxide (10.2%). There is also a minority presence of magnesium oxide (2.1%) manganese oxide (0.6%) and calcium oxide (0.4%) as shown Table 1 (Kpannieu et al., 2018).

2.2. Micro Pilot-Scale Unit Description

The experimental device that is the subject of this study was installed in the compound of the University Nangui Abrogoua (UNA) (Abidjan, Côte d'Ivoire). The pilots were designed from rectangular polyester tanks (Figure 1) with a volume of 14.8 L (L = 42 cm, l = 22 cm, H = 16 cm) as shown Figure 1. They were composed, from the bottom to the top, of layers of gravel (15/25 mm) which serve as the drainage material and the shale serves as the filtration material with respective thicknesses of 2 cm and 14 cm, shale granulometry (1/2 mm). These two massifs are separated by a geotextile which prevents obstruction of the drainage mass by shale. A filter is planted with young stems of *P. maximum* (20 stems/m^2) taken from mature and unplanted as controls as shown Figure 2. The pilot filters were fed with raw influent from a municipal wastewater in the Abobo Dokui district (Abidjan, Côte d'Ivoire). This wastewater is supplied to the filters by a PVC pipe system after the valves have been opened. Shale with porosity of 50% occupies about 14 L for 17 (kg) in the filter. A valve system under load ensures homogeneous distribution of applied wastewater. Thus, when the water reaches the height of 16 cm (bottom to top) of the tap, 7 L of water, the first drops begin to flow. The filters were intermittently fed with municipal Wastewater (MW) three times a week (Monday, Wednesday and Friday) between 3 pm and 4 pm the volume of MW applied to each filter during the feeding periods
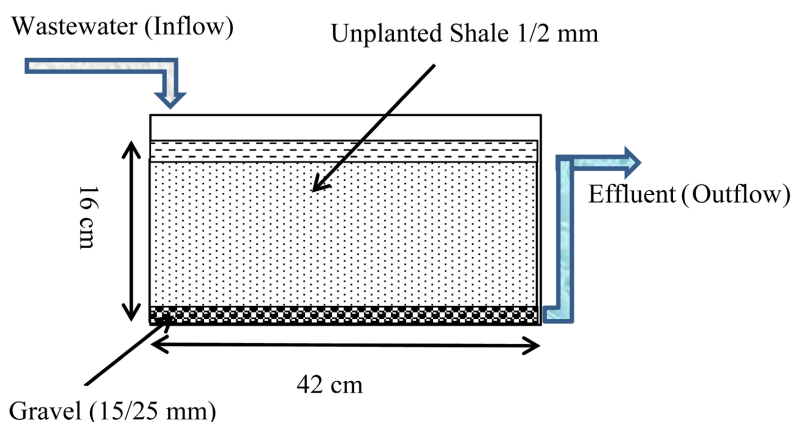

Figure 1. Schematic diagram of micro-pilot scale saturated vertical flow constructed wetland.

Figure 2. View of the wetland tanks.

is 7 L i.e. a flow rate of 3 L·J⁻¹ or a hydraulic load of 3.2 cm·J⁻¹. The hydraulic retention time (HRT) is 52 h. The experiment lasted nearly 87 days (February to June).

2.3. Water Quality Monitoring

During the treatment trials water samples were collected in 0.500 mL polyethylene bottles at the inlet (wastewater) and at the outlet (treated wastewater) from each filter every week and samples were refrigerated at 4°C until analyzed. Temperature, pH, redox potential and conductivity were determined in situ using a WTW multi-parameter WTW Multi 197i combination electrode. The Chemical Oxygen Demand (COD) is the amount of oxygen required for the oxidation of most of the organic material and some oxidizable inorganic ions (S^{2-}, Fe^{2+}, Mn^{2+}, etc.). The COD is determined by potassium dichromate oxidation in an acidic medium in accordance with the AFNOR T-90-101 standard. The 5-day Biological Oxygen Demand (BOD_5) is an indicator of pollution of the biodegradable organic matter. It represents the amount of oxygen used by the bacteria to partially decompose or to completely oxidize the biochemical oxidizable materials present in the water and which constitute their carbon source (fats, carbohydrates, surfactants…). It is determined by the manometric method with Oxitop WTW manometers according to the AFNOR T 90-103 standard. Total suspended solids (TSS) are obtained by vacuum filtration on a GF/C glass microfiber filter in accordance with the French standard AFNOR T 90-105. The concentrations ammonium, phosphate ($P\text{-}PO_4^{3-}$) and Total Phosphorus (TP) are determined by colorimetric methods. A spectrophotometer DR/2010 of HACH LANGE was used for Kjeldahl nitrogen (TKN) the titrimetric method in accordance with the French standard AFNOR T 90-11.

The percentage removal efficiency (R) was calculated as:

$$R = \left(\frac{C_i - C_t}{C_i} \right) \times 100 \qquad (1)$$

where C_i and C_t are inflow and outflow of contaminants concentrations.

3. Results and Discussion

3.1. Hydrodynamic Test

Table 2 presents some parameters determined during the hydrodynamic test

Table 2. Infiltration flow, volume of water returned and clogging rate during the hydrodynamic test carried out on filters planted with 20 stems m^{-2} with *Panicum maximum* (SCH1-2.P) and unplanted filter (control).

Filter	Before wastewater treatment		After wastewater treatment		
	Infiltration flow (mL·s^{-1})	Volume of water returned (mL)	Infiltration flow (mL·s^{-1})	Volume of water returned (mL)	Clogging rate (%)
Control	2.9	1000	1.9	650	34.5
SCH1-2.P	3.2	900	3	1000	6.2

carried out on the filter planted with *Panicum maximum* and on the non-planted control (shale of granulometry 1 - 2 mm) before and after 3 months of application of ERU. There is a decrease in the infiltration rate of water in the filters after application of ERU. This could be due to the clogging of the pores by the coarse and colloidal materials of the wastewater. In addition, it is found that the infiltration rate after wastewater treatment decreases much more significantly in the control than in the planted filter as shown Table 2. The feeding of the filters in wastewater induces the formation of a deposit which reduces the permeability of the solid mass as well as the transfer of oxygen into the filter. However, the growth of roots and rhizomes makes it possible to increase the permeability of planted filters, thus limiting the effect of the clogging layer. Indeed, the roots and stems of the plants pierce this layer and release free spaces to the flow around them, which increases the permeability of the massif (Kantawanichkul et al., 2003; Molle et al., 2011). This hypothesis is confirmed by the clogging rates determined after 3 months of 34% and 6.25% for the control filter and planted filter at 20 m^{-2} stems (SCH1-2.P) respectively. The presence of plants therefore limits the clogging process of the constructed wetland system.

3.2. Temperature and pH

Temperature and pH are important physical parameters to determine the water quality. In the present study, no significant temperature variation is known for municipal wastewater (influent) before and after treatment according to (Figure 3(a)). This observation particularly disagrees with the work of Brix (1994) in which planted filters have a lower temperature than unplanted filters. Plants shading limiting the penetration of ultraviolet rays, are provided to maintain the condition of freshness and humidity. The small size of the pilot in the study could explain difference obtained in results. However, one observes that the planted filters lead to a decrease in the pH of the municipal wastewater as shown (Figure 3(b)). According to WHO (2003) standards, the pH of water should lie in between 6.5 and 8.5. The pH of collected wastewater under study was 7.9. After subjected to wastewater treatment, pH of the planted as well as unplanted filters lied in a narrow acceptable range of 7.1 - 7.3. This slight decrease in pH
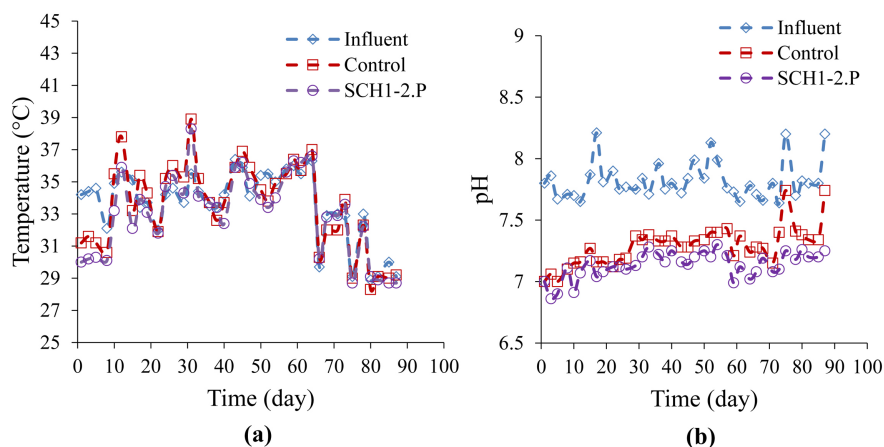
Figure 3. Variation in the temperature (a) and pH (b) of municipal wastewater (influent) after treatment through constructed wetlands with vertical-flow planted with *P. maximun* (SCH1-2.P) and unplanted (control) according to time.

might be due to the metabolism of sulphate, phosphate and nitrogenous compounds but also to the presence of *Panicum maximum* combined with shale which slightly accentuates the decrease in pH compared to the control. What is more, this decrease in pH is due to the respiration of plants or the degradation of the organic substance by heterotrophic bacteria (Coulibaly, 2008a, 2008b). Finally, the secretion of exudates (organic acids) from the plant roots can also contribute to the decrease of pH (Brix, 1997; Zhao et al., 2011).

3.3. TSS, COD and BOD

Figure 4 shows the profile of TSS, COD and BOD5 concentrations in influent and effluents. There is a very strong decrease in the concentrations of these pollutants from influent to effluent (**Figure 4(a)**). The reduction in TSS concentration is related to the filtering action of shale. It is also due to the interaction between microorganisms and dissolved oxygen, aerobic conditions of filters being favorable to the reduction of the organic matter. This degradation of organic matter may also be related to the effect of high temperature (Rivera et al., 1995; Chazarenc et al., 2011). With respect to planted filter, the decrease in TSS probably results from the symbiotic plant-bacteria relationship, in which bacteria use the oxygen supplied from the environment by plants during photosynthesis to degrade organic carbon (Kadlec & Knight, 1996). Moreover, according to Shama et al. (2015) the reduction of TSS of planted filters compared to the control is related to the mechanical action of the plant stems which increases the porosity of the massif and facilitates the flow of TSS. This assumption is not verified in the present study, because the very close TSS reduction rates determined for the planted filters and control. Here again the small size of the filters probably explains the difference of results observed. The removal ratio in COD and BOD$_5$ are high as shown (**Table 3**) and in good agreement with those obtained for a vertical-flow cws treating domestic wastewater with sand and gravel as a filter
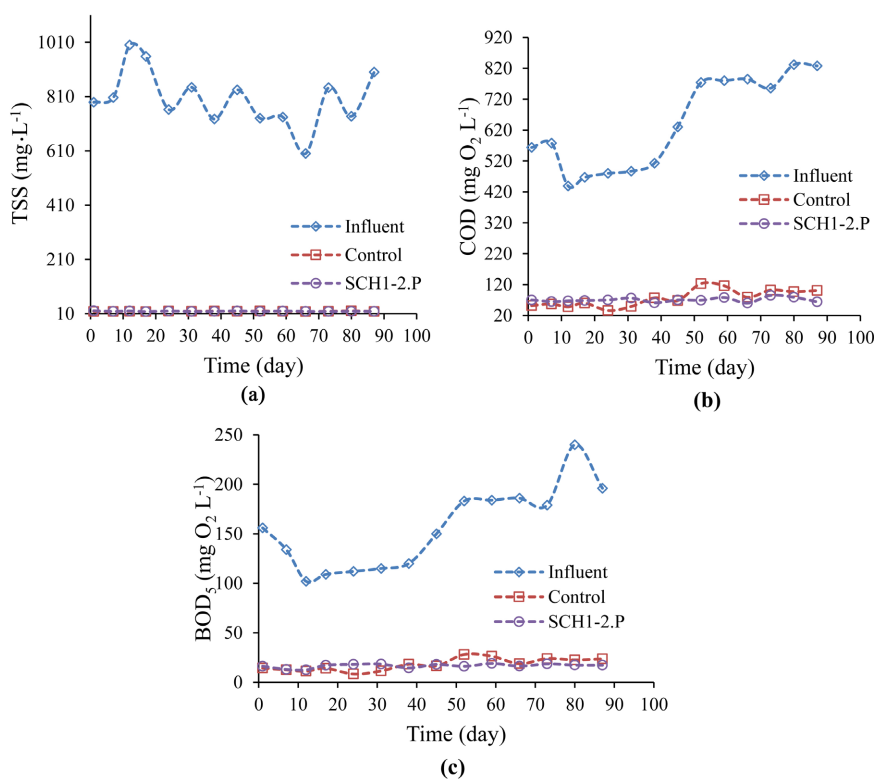
Figure 4. Variation in the TSS (a), COD (b) and BOD (c) of municipal wastewater (Influent) after treatment through constructed wetlands with vertical-flow planted with *P. maximun* (SCH1-2.P) and unplanted (control) at according to time.

Table 3. The effect of treatment of the micro-pilot scale on TSS, COD and BOD_5.

	Influent	Control		SCH1-2.P	
	Average value	Average value	Removal ratio (%)	Average value	Removal ratio (%)
TSS (mg·L^{-1})	804 ± 103	18.5 ± 0.7	98.0	18.7 ± 0.8	97.6
COD (mg O_2 L^{-1})	637 ± 149	66.4 ± 16.7	88.0	70.1 ± 7.1	89.4
BOD_5 (mg O_2 L^{-1})	154.7 ± 421.2	17.9 ± 6.7	88.0	16.4 ± 2	89.4

bed (Shama et al., 2015). The decrease in COD and BOD_5 and hence in organic matter is attributable to the microbial interaction and to the good aeration of the filter bed (Akratos & Tsihrintzis, 2007). The micro-pilot scale of this study (vertical flow cws) complies with the standards imposed by the European Directive 271/91/EEC of 21 May 1991, that is to say concentrations lower than 35 mg TSS L^{-1}; 125 mg COD L^{-1} and 25 mg BOD_5 L^{-1}.

3.4. Nitrogen Removal: Total Kjeldahl Nitrogen (NTK) and Ammonium Ions (NH_4^+)

The concentrations of total kjeldahl nitrogen (NTK) and ammonium ions (NH_4^+) in the municipal wastewater (influent) and in the effluents are shown in **Figure**

5. There is a significant decrease in nitrogen concentrations in the effluents compared to the influent. The removal ratio for NTK nitrogen is substantially the same regardless of the filter considered as shown in (Table 4). The ammonium ion removal ratio of the planted filter (84%) is slightly higher than the control (79%) (Table 4). The high rate of removal of NTK nitrogen and NH_4^+ ions is probably related to the high temperature. Christos and Vassilios (2007) particularly showed higher removal rates of NTK nitrogen and NH_4^+ ions at temperatures above 15°C in a CWs made of sand and gravel. In general, high temperatures result in higher biological activity and growth rates of the microorganisms as well as volatilization of ammonia, hence a higher removal rate of NTK [2]nitrogen and NH_4^+ ions (Faulwetter et al., 2009; Truu et al., 2009; Tanner et al., 1999). It is important to note that shale adsorbs NTK nitrogen and NH_4^+ ions as previously described (Drizo, 1997), which may also explain the decrease of these pollutants in the filtrates. However, nitrification-denitrification processes are commonly considered as the major mechanisms for nitrogen removal in artificial wetlands (Brix & Schierup, 1990; Tanner et al., 1999).
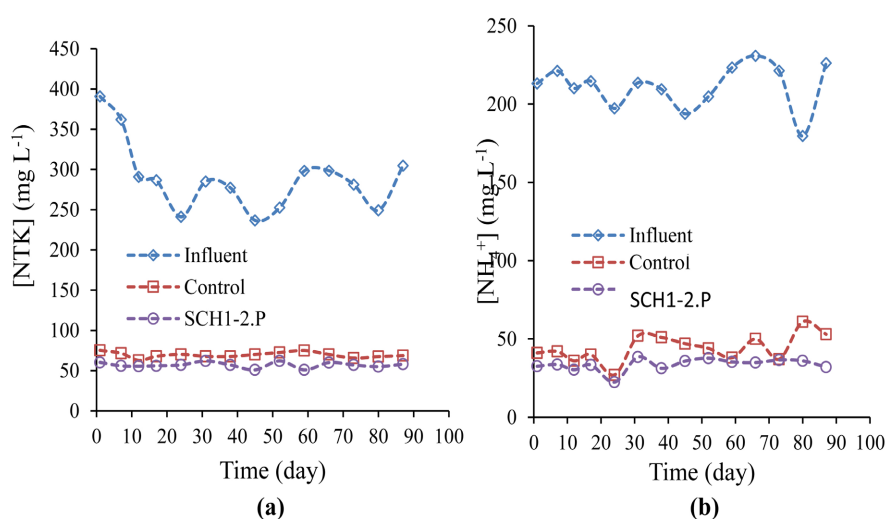

Figure 5. Variation in the TSS (a) and NH_4^+ (b) of municipal wastewater (Influent) after treatment through constructed wetlands with vertical-flow planted with *P. maximun* (SCH1-2.P) and unplanted (control) according to time.

Table 4. The effect of treatment of the micro-pilot scale on NTK and NH_4^+.

	Influent	Control		SCH1-2.P	
	Average value	Average value	Removal ratio (%)	Average value	Removal ratio (%)
NTK (mg·L^{-1})	290 ± 43	68.7 ± 3.2	76.0	57.0 ± 3.4	80.0
NH_4^+ (mg·L^{-1})	211.4 ± 13.0	44.2 ± 8.7	79.0	33.6 ± 4.0	84.0

3.5. Phosphorus Removal: Total Phosphorus (TP) and Orthophosphate Ions ($P-PO_4^{3-}$)

As illustrated in **Figure 6** it is noted that the TP concentrations are significantly lower in the filtrates than in the municipal wastewater (**Figure 6(a)**). The average removal rate obtained is substantially the same for the planted filter and the control as shown in (**Table 5**). The adsorption capacities (qp) are ~0.2 mg·g⁻¹ for the two filters. The adsorption capacity obtained exclusively by the plants that is to say the difference between the adsorption capacity of the planted filters and the control is very low (~0.02 mg·g⁻¹) and reinforces the need research of reactive materials for planted filters. The concentration of $P-PO_4^{3-}$ decreases considerably in the filtrates compared to the influent (**Figure 6(b)**). The removal rates are in the following order: SCH1-2 P (92%) > control (86%) as shown in (**Table 5**). The adsorption capacity as for it is practically identical and
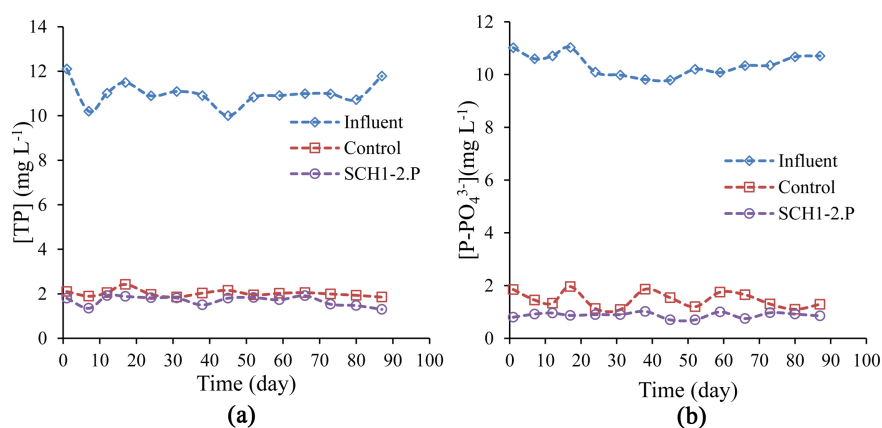

Figure 6. Variation in the TP (a) and $P-PO_4^{3-}$ (b) of municipal wastewater (Influent) after treatment through constructed wetlands with vertical-flow planted with *P. maximun* (SCH1-2.P) and unplanted (control) according to time.

Table 5. The effect of treatment of the micro-pilot scale on TP and $P-PO_4^{3-}$.

	Influent	Control		SCH1-2.P	
	Average value	Average value	Removal ratio (%)	Average value	Removal ratio (%)
TP (mg·L⁻¹)	11.01 ± 0.5	2.0 ± 0.1	82.0	1.8 ± 0.3	83.8
$P-PO_4^{3-}$ (mg·L⁻¹)	10.38 ± 0.41	1.5 ± 0.3	86.0	0.9 ± 0.1	92.0

close to ~0.2 mg·g⁻¹ of P-PO₄ for the two filters. However, as before, the exclusive adsorption capacity of the plants is very low. TP and $P-PO_4^{3-}$ may in particular be eliminated according to physical mechanisms (accumulation on the surface of the filter), physicochemical (adsorption and precipitation), biological (plant assimilation and microorganisms). Other studies have shown superior re-

moval rate in the case of planted filters (Fonkou et al., 2011; Vymazal, 1995). Indeed, phosphorus is assimilated by plants by adsorption by the root system and accumulates in the leaves and young shoots for their growth. Cutting the aerial part of growing plants allows the removal of phosphorus (IWA, 2000). As previously, the difference in results could be explained by the small size of the pilot, which does not make it possible to highlight the influence of the density of plants. However, the exclusive use of plants for the treatment of phosphorus is not economically viable because too large areas of treatment with population equivalent (PE) would be required. This hypothesis has been corroborated by the results of the present study. The use of reactive materials for the retention of phosphorus on a solid phase by adsorption and/or precipitation mechanisms is a very promising alternative (Vohla et al., 2011). The results presented in this study support this hypothesis, since more than 86% of phosphorus pollution is removed by the filter control. In addition, throughout the duration of the test, the filter is not saturated. This result is consistent with the work of Drizo et al. (1997) in which shale was also used as a filter bed for a CWs.

3.6. Technical Study of a CWs Implementing Shale on a Large Scale

Let us consider a given quantity of wastewater containing a phosphate pollution of population equivalent (PE), each PE producing approximately 3 g of phosphate per day (Johansson, 2006; Li et al., 2008). The total quantity of phosphate that should be adsorbed in four years represents a total mass 4.38 kg PE^{-1}. Moreover, if one considers that the passive treatment (CWs) should last at least 4 years before the shale would be replaced, the adsorption capacity of the micro pilot scale is ~0.2 mg·g^{-1}. Therefore, as mass of ~21.9 tons PE^{-1} of shale would be necessary and the corresponding volume of the reactor would be ~24.97 m^3 PE^{-1} since the apparent density of shale is ~1.14 as shown in (Table 1). Considering a Saturated Vertical-flow Constructed Wetlands on 0.5 depth, this volume corresponds to a surface of ~38 m^2 PE^{-1} (Truong et al., 2011). The CWs will have a relatively large surface area. This large surface area may not be a problem for developing countries such as Côte d'Ivoire, given their low industrialization and therefore low land use in these countries. In addition, in order to increase the sustainability of the process with natural materials, as well as its economic viability, the recovery routes in fine phosphorus saturated material and plant biomass should be considered.

4. Conclusion

Saturated Vertical-flow Constructed Wetlands (SVCWs) using shale of Côte d'Ivoire as a substrate of this study were efficient in removing TSS (98%), DOC (89.4%), BOD$_5$ (89.4%), NH$_4^+$ (84%), TKN (80%), TP (84.8) and P-PO$_4^{3-}$ (92%). The adsorption capacity of phosphate ions was 0.2 mg without the shale being saturated in phosphorus during the period of study. Shale has therefore demon-

strated its ability to remove phosphorus and nitrogen pollution from wastewater. The use of this system can help reduce eutrophication effects in receiving streams and improve water quality. More research is needed to enhance the efficiency of constructed wetlands for over-whelming the problem of water pollution in developing countries.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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