Journal of Water Resource and Protection, 2025, 17(1), 14-34
https://www.scirp.org/journal/jwarp
ISSN Online: 1945-3108
ISSN Print: 1945-3094

Optimizing the Particle Size of Shale and Laterite Used as Constructed Wetland Substrates for Wastewater Treatment

Nadège Fatim Traoré[1], Jean-Marie Pétémanagnan Ouattara[1], Franck Michaël Zahui[2*], Amichalé Jean Cyrille Beda[2], Aman Messou[1]

[1]Department of Sciences and Environment Management, Nangui Abrogoua University, Abidjan, Côte d'Ivoire
[2]Department of Agronomic, Forestry and Environmental Engineering, University of Man, Man, Côte d'Ivoire
Email: *michael.zahui@univ-man.edu.ci

How to cite this paper: Traoré, N.F., Ouattara, J.-M.P., Zahui, F.M., Beda, A.J.C. and Messou, A. (2025) Optimizing the Particle Size of Shale and Laterite Used as Constructed Wetland Substrates for Wastewater Treatment. *Journal of Water Resource and Protection*, **17**, 14-34.
https://doi.org/10.4236/jwarp.2025.171002

Received: December 4, 2024
Accepted: January 6, 2025
Published: January 9, 2025

Abstract

Constructed Wetlands (CWs) are currently one of the most promising techniques for wastewater treatment, having demonstrated their effectiveness. However, the choice of substrate particle size is critical to the smooth operation of the process, as hydrodynamic constraints require a coarse particle size, whereas wastewater treatment recommends a fine particle size. This study investigates the suitability of laterite and shale as substrates of different sizes (1 - 3, 3 - 5 and 5 - 8 mm) in CWs for domestic wastewater treatment. The study was carried out in an experimental pilot plant consisting of 12 parallelepiped beds ($C \times C = 0.4 \times 0.4$ m^2; H = 0.6 m) filled from bottom to top with 0.1 m of gravel and 0.4 m of shale or laterite of different grain sizes with two replications. During the six months of operation, plant biomass and stem diameter of *Pennisetum purpureum* used as vegetation in the CWs were determined. Raw and treated water were also sampled and analyzed for pollutants, including chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), total Kjedahl nitrogen (TKN), total phosphorus (TP), and total suspended solids (TSS), using International Organization for Standardization (ISO) analytical methods. *P. purpureum* developed much better in the CW beds lined with shale; plant biomass ranged from 13.8 to 14.7 kg/m^2 and from 11.2 to 12.5 kg/m^2 in the beds lined with shale and laterite, respectively, as did stump diameter, which ranged from 15.5 to 16.1 cm and from 11.10 to 12.7 cm, respectively. However, the highest values for biomass and stump diameter for each material were obtained in the beds lined with 1 - 3 mm geomaterials. Pollutant removal efficiencies were highest in the CWs lined with laterite and shale of 1 - 3 mm grain size (76.9% - 83% COD, 78% - 84.7% BOD_5, 55.5% - 72.2% TKN, 58.4% - 72.4% TP, 78.1% - 80.2% TSS), with the highest values recorded in the shale-lined beds. However, the 3 - 5 mm grain size of both materials provided quality filtrates (140 - 174 mg/L COD, 78.5 - 94.8 mg/L BOD5, 4.6 - 5.7 mg/L TP) in line with local wastewater discharge levels. This size of geomaterials appears to be suitable for optimization purposes, although further work with these materials, such as increasing the depth of the wetland, is required to improve the level of NTK and TSS discharge.

Keywords

Constructed Wetlands, Domestic Wastewater, Laterite, *Pennisetum purpureum*, Shale, Substrate Grain Size

1. Introduction

Constructed wetlands (CWs), also known as artificial wetlands, are currently one of the most promising wastewater treatment technologies, having demonstrated their effectiveness in removing pollutants from a wide range of wastewater types (domestic, industrial, landfill, etc.) for decades [1]-[3]. CWs are also known for their relatively low installation and operating costs and energy consumption compared to conventional wastewater treatment technologies such as activated sludge, biological discs, etc. [4] [5]. Furthermore, in addition to the potential of CWs to offset energy demand (through the use of plant biomass) and water demand in agriculture (through the use of treated wastewater for irrigation) [6], artificial wetlands have an asset related to the aesthetics of the landscape in which they are installed or located [7]-[9].

The treatment of wastewater in constructed wetlands is carried out by physical, chemical and biological mechanisms and in all cases involves the components of CWs: the plant, the micro-organisms and the filtration material that forms the substrate for the first two. While the microorganisms develop naturally in the substrate of the constructed wetland during its operation, the plant and the filtration material are selected by the manufacturer during the implementation of the process [2] [10]. However, the contribution of plants used in constructed wetlands to the process of reducing the pollutant load of wastewater is both direct and indirect in the wastewater treatment process. Plants have the capacity to assimilate nutrients (nitrogen and phosphorus) from wastewater for their metabolism and/or to store them. However, several studies seem to show that plants play a minimal role in the direct removal of nutrients by assimilation. However, some plants secrete antibiotics and use these metabolites to help eliminate pathogenic microorganisms from wastewater. The indirect contribution of plants to the purification process can be seen in the stabilization of the substrate by the root system, the maintenance of flow capacity, the creation of a microclimate and the oxygenation of the substrate, all of which are conducive to the establishment of periphyton and purifying microorganisms. However, in addition to adaptation to climatic conditions, resistance to anoxic and hyper-eutrophic situations and the ability to absorb pollutants are suggested for the selection of plants in constructed wetlands [2] [7] [11] [12].

Due to their particle size, substrates play a role in filtering suspended solids (SS) present in wastewater and provide, among other things, a physical support for plants and a reactive surface for the transformation of chemical elements, as well as an ecosystem for microbial fauna and/or macrofauna in the constructed wetlands [10]. The chemical constituents of the substrate (e.g. iron, aluminium, etc.) influence the chemical reactions that take place there in the CWs. Therefore, the selection of the substrate based on, among other things, its potential to absorb pollutants from the wastewater to be treated seems to be an added value to the wastewater treatment process using constructed wetlands (Vymazal, 2022) [2]. Several research reviews, including those by Sanjrani *et al.* [13], Rahman *et al.* [10], Ji *et al.* [14], Sandoval Herazo *et al.* [15] and Wang *et al.* [16] on substrates based on natural mineral materials commonly used in constructed wetlands mention sand, gravel, clay, calcite, marble, bentonite, dolomite, limestone, shell, zeolite, peat and others. However, geological materials such as shale and laterite remain largely unexplored, although they are abundant in some areas and their mineralogical composition gives them adsorptive properties that could be put to good use in the purification performance of constructed wetlands. In addition to the kaolinite present in shale and laterite, the iron and alumina oxides and hydroxides present in laterite and the other forms of clay (albite and dolomite) present in shale enable them to bind the nitrogen and phosphorus compounds present in wastewater [17] [18].

However, while hydrodynamic constraints (*i.e.* adequate flow rate) require a coarse particle size for the filtration material, wastewater treatment requires a fine particle size; this remains a major trade-off to be considered when selecting filtration material for constructed wetlands [19] [20]. Therefore, the choice of particle size for the filtration material has to fulfil a double objective: to ensure the effectiveness of the wastewater treatment and to guarantee the lifetime of the constructed wetlands. This has led to several studies, including those by Zhao *et al.* [21] [using zeolites, gravel and anthracites] and [22] [using ceramics], among others. Relative to shale and laterite, these geomaterials have been successfully tested simultaneously for the treatment of domestic wastewater in constructed wetlands due to their availability and abundance [23]. However, optimizing the granulometry of these materials, taking into account only compliance with the effluent discharge limits into the receiving environment, would reduce the effort and resources required to crush these materials. This is because fine-grained materials require larger quantities of geomaterials to be crushed than coarser-grained materials [24]. Similarly, fine-grained materials are more prone to rapid clogging of constructed wetlands, which reduces the lifetime of the wetland, even though the treatment of wastewater pollutants is more advanced in this type of material [19] [25] [26].

This study investigates the influence of the grain size of shale and laterite (used as substrates) on the operation of constructed wetlands for the treatment of domestic wastewater. The objectives are to determine the purification performance of Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD_5), Total Nitrogen Kjedahl (TNK), Total Phosphorus (TP) and Total Suspended Solids (TSS) in constructed wetlands equipped with laterite and shale of different grain sizes (1 - 3, 3 - 5 and 5 - 8 mm), to access the compliance of wetland effluent pollutant concentrations with wastewater discharge levels to the receiving environment, and to investigate the response of plant growth in the constructed wetlands (*i.e.* above ground biomass and plant stump diameter).

2. Methods

2.1. Experimental Setup

The study was carried out at the experimental pilot plant previously described by Traoré *et al.* [23] at the Nangui Abrogoua University, Abidjan, Côte d'Ivoire. The site is characterized by a humid tropical climate with an average temperature of 25 to 33.2 C and an average rainfall of 23 to 525 mm [27]. The experimental set-up consisted of twelve (12) beds in the shape of a square-based right block ($C \times C = 0.4 \times 0.4\ m^2$, with $H = 0.6$ m), six (6) of which were filled with the same geomaterials (*i.e.* shale or laterite). The beds were filled from bottom to top with a 0.1 m layer of gravel (5/25 mm) and a 0.4 m layer of substrate consisting of shale or laterite of different grain sizes (1 - 3, 3 - 5 and 5 - 8 mm), separated by a geotextile. Each bed was equipped with an outlet ($\Phi = 32$ mm) to drain the percolation water (treated water).

The beds were planted with young *Pennisetum purpureum* plants (20 cm tall) from a nursery established for this purpose at the experimental site. Three (3) three-week-old plants of the same vigor were transplanted into each reactor, with a spacing of 30 cm between plants, relatively similar to that used by Zahui *et al.* [28]. The lilies were fed for three days a week (*i.e.* Monday, Wednesday and Friday) with raw domestic wastewater ($6.43\ L \cdot d^{-1}$), collected from the Nangui Abrogoua University wastewater network and stored in a cubitainers ($1\ m^3$) 1.5 m above ground, from which the beds were fed (**Figure 1**).

2.2. Preparing Substrates for Constructed Wetlands

Laterite and shale blocks crushed for use as substrates in constructed wetlands were collected from the Lomo-Nord site at Toumodi in central Côte d'Ivoire (6˚39'0''N 4˚58'60''W). The samples were washed and steamed, then separately crushed with a hammer and sieved through a series of sieves (**Figure A1**). Granular fractions with diameters of 1 - 3 mm, 3 - 5 mm and 5 - 8 mm (**Table 1**) were collected for the processing tests. Thus, the choice of particle sizes was based on literature data [29] [30]. Once the granular fractions were obtained, they were washed to remove any impurities before being placed in the various artificial wetland beds. The mineralogical analysis of these geomaterials shows that they are rich in Al and Fe, with respectively 49.9% Fe_2O_3 and 30% Al_2O_3 for the shale and 31.40% Fe_2O_3 and 66.01% Al_2O_3 for the laterite.

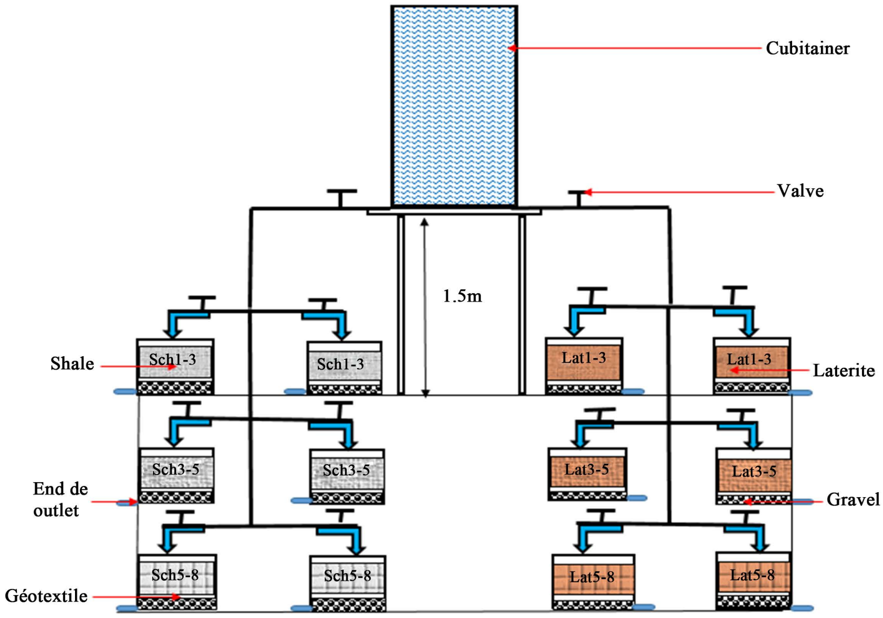

Figure 1. Schematic of the experimental pilot.

Table 1. Raw geomaterials and crushed geomaterials with a range of grain sizes for use in experiments.

	Raw geomaterials	Crushed geomaterials	
Laterite		1 - 3 mm	
		3 - 5 mm	
		5 - 8 mm	
Shale		1 - 3 mm	
		3 - 5 mm	
		5 - 8 mm	

2.3. Monitoring *Pennisetum purpureum* Growth in Constructed Wetland Beds

Plant growth in the constructed wetland beds was monitored by measuring the diameter of the plant stumps and the plant biomass produced. The plants were harvested at the end of the two-month growth cycle of the P purpureum stems and the diameter of the plant stumps was measured to the millimeter during mowing. A total of three plant harvests were made, during which the production of fresh plant biomass was determined after weighing the harvested biomass according to Equation (1)

$$P = \frac{FB}{S} \tag{1}$$

with: *P*: production (kg/ m^2), *FB*: fresh biomass (kg) and *S*: surface of the bed (m^2).

2.4. Water Sampling and Analysis of Physico-Chemical Parameters

Water samples were taken at the inlet and outlet of the constructed wetland beds at regular intervals of 15 days and stored in polyethylene bottles at 4˚C until analysis, *i.e.* 12 samples at the inlet and outlet of each bed. Physical parameters such as pH, dissolved oxygen (DO), electrical conductivity (EC) and suspended solids (SS) were determined according to ISO 10523 [31], ISO 5814 [32], ISO 7888 [33] and ISO 11923 [34] respectively. As for the analysis of chemical parameters, Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD_5), Total Nitrogen Kjedahl (TNK) and Total Phosphide (TP) were determined according to ISO 6060/2 [35], ISO 5815/1 [36], ISO 5663 [37] and ISO 6878/2 [38] respectively.

Based on the concentrations of the above-mentioned pollutants (*i.e.* COD, BOD5, NTK, TP and TSS), the purification efficiencies of the wetlands were calculated according to relationship 2 described by Abissy and Mandi [39].

$$\text{Purification efficiency}(\%) = \frac{\text{Input load}(\text{mg}) - \text{Output load}(\text{mg})}{\text{Input load}(\text{mg})} \times 100 \tag{2}$$

with:

Input load = concentration (mg/L) × volume of raw wastewater (L) fed to the bed;

Output load = concentration (mg/L) × volume of water (L) returned at the outlet of the bed.

2.5. Data Analysis

In order to analyze the data, statistical tests were carried out to compare the data from the beds of the different constructed wetlands (*i.e.* biomass production, plant stump diameter, pollutant concentrations, wetland purification efficiencies, volumes of water returned at the outlet of the beds, etc.). The tests used were the Shapiro-Wilk test to determine the normality of the data, followed by the ANOVA and T-test in the case of a normal distribution of the data, or the Kruskal-Wallis test followed by the Mann-Whitney test, depending on whether the data followed a non-normal distribution. In all cases, the difference was considered significant when $p < 0.05$. These statistical tests were performed using R studio 3.3.2 software.

3. Results

3.1. Plant Growth Response

Figure 2 shows the plant biomass and stump diameter developed by *Pennisetum purpureum* in beds filled with shale and laterite during the experiment. Overall, the plant biomass and stump diameter of the plants increased as the grain size of the geomaterials decreased, regardless of the geomaterials. However, the plant biomass and stump diameter measured in the shale beds were higher than those in the laterite beds. As shown in Figure 2(A), the biomasses were 14.7 ± 0.5 kg/m^2, 14.1 ± 0.5 kg/m^2 and 13.8 ± 0.3 kg/m^2 in beds filled with shale of grain sizes 1 - 3, 3 - 5 and 5 - 8 mm, respectively, and 12.5 ± 0.1 kg/m^2, 11.9 ± 0.2 kg/m^2 and 11.2 ± 1 kg/m^2 in beds filled with laterite of grain sizes 1 - 3, 3 - 5 and 5 - 8 mm, respectively. Figure A2 shows plant growth in the beds of constructed wetlands.

As for the stump diameter (Figure 2(B)), the measurements taken were 16.1 ± 1.3 cm, 15.5 ± 1.4 cm and 14 ± 1.5 cm in the beds filled with shale of grain sizes 1 - 3, 3 - 5 and 5 - 8 mm, respectively, and 12.7 ± 1.4 cm, 11.9 ± 1.4 cm and 11.1 ± 1.8 cm in the beds filled with laterite of grain sizes 1 - 3, 3 - 5 and 5 - 8 mm, respectively. Statistical analysis showed that the biomass and stump diameter of the plants produced in the constructed wetlands lined with shale were significantly higher than those in the wetlands lined with laterite, considering the homologous grain sizes (T-test: $p < 0.05$). On the other hand, there was no significant difference when comparing plant biomass and stump diameter measured in beds of the same geomaterials (shale or laterite) (T-test: $p > 0.05$).

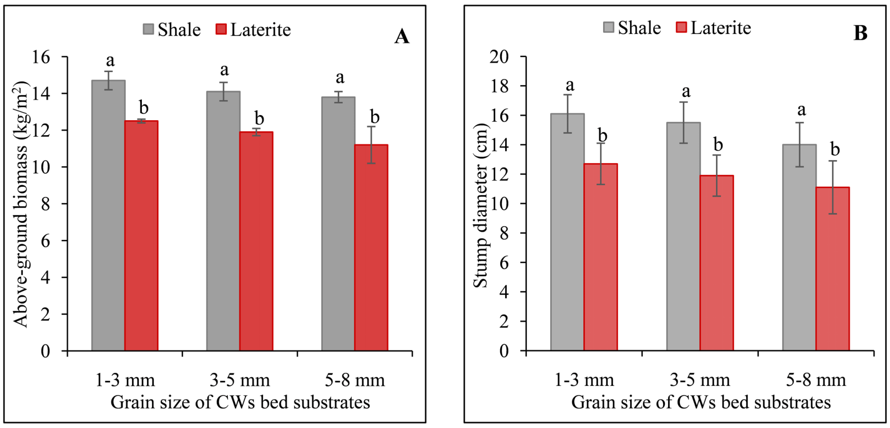

Figure 2. Biomass (A) and stump diameter (B) of *Pennisetum purpureum* in constructed wetlands lined with slate laterite with grain sizes of 1 - 3, 3 - 5 and 5 - 8 mm, respectively. Histogram with same letter indicates no significant difference ($p > 0.05$).

3.2. Wastewater Treatment Performance

3.2.1. Physical Parameters

Table 2 shows the minimum, maximum and average values of effluent volume, pH, dissolved oxygen (DO) and electrical conductivity (EC) measured at the inlet and outlet of wetland beds lined with shale and laterite of grain sizes 1 - 3 mm (Sch1-3, Lat1-3), 3 - 5 mm (Sch3-5, Lat3-5) and 5 - 8 mm (Sch5-8, Lat5-8). Overall, the volume of water returned at the outlet of the wetland beds was significantly less than the volume of effluent applied [Mann-Whitney test: $p < 0.05$]. However, the volumes of water collected at the outlet of the laterite filled beds (13.63 - 14.42 liters) were higher than those of the shale filled beds (12.96 - 14.40 liters). However, for beds lined with the same material (shale or laterite), the volume of water returned increased with the grain size of the geomaterials. The same was true for electrical conductivity, for which 451 $\mu S \cdot cm^{-1}$ (Sch1-3), 478 $\mu S \cdot cm^{-1}$ (Sch3-5) and 527 $\mu S \cdot cm^{-1}$ (Sch5-8) were measured at the outlet of beds lined with shale, and 479 $\mu S \cdot cm^{-1}$ (Lat1-3), 519 $\mu S \cdot cm^{-1}$ (Lat3-5) and 569 $\mu S \cdot cm^{-1}$ (Lat5-8) at the outlet of beds lined with laterite.

On the other hand, the values measured for dissolved oxygen were higher at the outlet of the beds than in the raw water and decreased with increasing geomaterials grain size (T-test: $p < 0.05$). For an average of 2 mg/L in the raw water, 7.2 mg/L (Sch1-3), 6.1 mg/L (Sch3-5) and 5.2 mg/L (Sch5-8) were recorded at the outlet of beds lined with shale, and 6.4 mg/L (Lat1-3), 5.4 mg/L (Lat3-5) and 4.6 mg/L (Lat5-8) at the outlet of beds lined with laterite. As for the pH, the average value recorded in the wastewater (7.9) fell sharply in the filtrates from the wetland beds (7.2 - 7.4) [T-test: $p < 0.05$]. However, the values varied very little from one geomaterials to another and from one grain size to another (T-test: $p > 0.05$).

Table 2. Minimum, maximum and average values for volumes of wastewater, pH, dissolved oxygen and electrical conductivity measured at the inlet (wastewater) and outlet (filtrates) of constructed wetlands.

Treatment and parameter			Volume (L)	CE (µS/cm)	OD (mg/L)	pH
Wastewater		Aver	15^{a}	1633 ± 94.7^{b}	2 ± 0.5^{b}	7.9 ± 0.4^{a}
		Max	-	1825	3	9.5
		Min	-	1446	1.1	6.8
Beds lined with shale	Sch1-3	Aver	12.96 ± 0.5^{b}	451 ± 48.7^{a}	7.2 ± 0.9^{a}	7.2 ± 0.3^{b}
		Max	14	577	9	7.8
		Min	12	342	4.8	6.4
	Sch3-5	Aver	13.29 ± 0.4^{b}	478 ± 54.1^{a}	6.1 ± 1^{ad}	7.4 ± 0.3^{b}
		Max	14	625	8	8
		Min	13	340	3.5	6.7
	Sch5-8	Aver	14.04 ± 0.1^{c}	527 ± 45.7a	5.2 ± 1.1^{d}	7.4 ± 0.3^{b}
		Max	14.5	681	7.5	7.8
		Min	13.5	441	2.9	6.6

Continued

Beds lined with laterite	Lat1-3	Aver	13.63 ± 0.5^{d}	479 ± 51.2^{a}	6.4 ± 1.1^{a}	7.3 ± 0.2^{b}
		Max	14.5	603	8.5	7.8
		Min	13	365	3.6	6.8
	Lat3-5	Aver	14 ± 0.1^{d}	519 ± 58.8^{a}	5.4 ± 1.2^{ac}	7.3 ± 0.3^{b}
		Max	14.5	689	7.2	7.8
		Min	13.5	379	2.6	6.5
	Lat5-8	Aver	14.42 ± 0.2^{e}	569 ± 42.8^{a}	4.6 ± 1^{c}	7.4 ± 0.3^{b}
		Max	14.5	701	6.3	7.8
		Min	14	483	2	6.1

Values in the same column followed by the same superscript letter (*i.e.* a, b, c...) indicate no significant difference ($p > 0.05$). Beds lined with shale (Sch1-3, Sch3-5 and Sch5-8) and laterite (Lat1-3, Lat3-5 and Lat5-8) with grain sizes of 1 - 3, 3 - 5 and 5 - 8 mm respectively.

3.2.2. Chemical Parameters

Table 3 shows the minimum, maximum, average and purification efficiency values for total suspended solids (TSS), chemical oxygen demand (COD) and biochemical oxygen demand (BOD_5), total Kjedahl nitrogen (TKN) and total phosphorus (TP) measured at the inlet (wastewater) and outlet (filtrates) of wetland beds lined with shale and laterite with grain sizes of 1 - 3 mm (Sch1-3, Lat1-3), 3 - 5 mm (Sch3-5, Lat3-5) and 5 - 8 mm (Sch5-8, Lat5-8). Overall, the TSS at the outlet of the wetland beds was significantly lower than that of the raw water (238.9 mg/L) [Mann-Whitney test: $p < 0.05$]. However, the average TSS concentrations obtained at the outlet of the laterite-lined beds (51.7 mg/L [Lat1-3], 73.7 mg/L [Lat3-5] and 86.3 mg/L [Lat5-8]) were higher than those of the shale-lined beds (48.7 mg/L [Sch1-3], 68.4 mg/L [Sch4-5] and 79.8 mg/L [Sch5-8]). However, for beds lined with the same material (shale or laterite), TSS concentrations increased with the grain size of the geomaterials. Thus, TSS removal rates decreased with increasing geomaterials grain size, with values ranging from 65.2 to 80.2% for beds lined with shale and from 61.2 to 78.1% for beds lined with laterite. The same was true for the chemical oxygen demand (COD), which was 481.3 mgO_2/L in the raw water, with average values of 92.4 mgO_2/L (Sch1-3), 140.5 mgO_2/L (Sch3-5) and 208.9 mgO_2/L (Sch5-8) measured at the outlet of the beds lined with shale, and 118.7 mgO_2/L (Lat1-3), 174.5 mgO_2/L (Lat3-5) and 267.7 mgO_2/L (Lat5-8) at the outlet of the beds lined with laterite. These corresponded to BOD_5 removal efficiencies of 58.5 to 83% in the shale-lined beds and 46.1 to 76.9% in the laterite-lined beds.

Like TSS and COD, the concentrations of biochemical oxygen demand measured were lower at the outlet of the beds than in the raw water, and decreased when the grain size of the geomaterials decreased (T-test: $p < 0.05$). For an average of 288.6 mgO_2/L in raw water, 48.3 mgO_2/L (Sch1-3), 78.5 mgO_2/L (Sch3-5) and 118.2 mgO_2/L (Sch5-8) were recorded at the outlet of beds lined with shale, and 65.4 mgO_2/L (Lat1-3), 94.8 mgO_2/L (Lat3-5) and 151.3 mgO_2/L (Lat5-8) at the outlet of beds lined with laterite. Equivalent BDO_5 purification efficiencies ranged from 60.2 to 84.7% in the shale-filled beds and from 48.2 to 78% in the laterite-filled beds. As for total Kjedahl nitrogen, the mean value recorded in the raw water (143.6 mg/L) decreased significantly in the wetland bed filtrates [Mann-Whitney: $p < 0.05$]. However, the NTK concentrations obtained at the outlet of laterite-filled beds (71.9 mg/L (Lat1-3), 81.5 mg/L (Lat3-5) and 92.6 mg/L (Lat5-8)) were higher than those of shale-filled beds (46.5 mg/L (Sch1-3), 66.2 mg/L (Sch3-5) and 78.3 mg/L (Sch5-8)). However, for beds filled with the same material (shale or laterite), NTK concentrations increased with the grain size of the geomaterials. This represented between 49.9 and 72.2% NTK removal in beds filled with shale and between 38.3 and 55.5% in beds filled with laterite. Like TSS, COD, BOD_5 and NTK, total phosphorus concentrations were lower at the bed outlet, and decreased as the grain size of the geomaterials decreased (T-test: $p < 0.05$). The mean values recorded indicated 11.8 mg/L in the raw water, 3.7 mg/L (Sch1-3), 4.6 mg/L (Sch3-5) and 5.5 mg/L (Sch5-8) at the outlet of the beds lined with shale and 5.4 mg/L (Lat1-3), 5.7 mg/L (Lat3-5) and 6.7 mg/L (Lat5-8) at the outlet of the beds lined with laterite. Total phosphorus purification efficiencies ranged from 56.3% to 72.4% in the shale-filled beds and from 44.7% to 58.4% in the laterite-filled beds. These yields, as for the above-mentioned parameters apart from NTK and TSS, differed significantly overall for each grain size. (T-test: $p < 0.05$). However, overall, the yields obtained by the beds filled with materials of grain size 1 - 3 mm were statistically different from the others ($p < 0.05$).

Table 3. Minimum, maximum and average concentrations determined in wastewater and in filtrates from beds lined with shale (Sch1-3, Sch3-5 and Sch5-8) and laterite (Lat1-3, Lat3-5 and Lat5-8).

Treatment and parameter			TSS		COD		BOD_5		TNK		TP	
			Value (mg/L)	R (%)	Value (mgO_2/L)	R (%)	Value (mgO_2/L)	R (%)	Value (mg/L)	R (%)	Value (mg/L)	R (%)
Wastewater		Aver	238.9^a	-	481.3^a	-	288.6^a	-	143.6^a	-	11.8^a	-
		Max	480	-	644.8	-	389.3	-	194.7	-	13.8	-
		Min	166.8	-	297.4	-	163.5	-	97.5	-	10.2	-
Beds lined with shale	Sch1-3	Aver	48.7^b	80.2^a	92.4^b	83^a	48.3^b	84.7^a	46.5^b	72.2^a	3.7^b	72.4^a
		Max	59.5	93.1	133.7	92.1	89.2	96.4	86.4	88.6	4.9	81.7
		Min	20.3	71.3	45.8	68.2	15.2	67	16.7	50	2.8	65.6
	Sch3-5	Aver	68.4^{cd}	71.5^b	140.5^c	73.1^b	78.5^c	74.1^b	66.2^b	60.6^{ba}	4.6^c	65.5^b
		Max	89	92	204.3	86.8	112.4	88.5	126.2	87	5.5	73.2
		Min	42.5	57.6	81.5	54.7	50.3	50.7	21	32.7	3.8	53.2
	Sch5-8	Aver	79.8^d	65.2^c	208.9^d	58.5^c	118.2^d	60.2^c	78.3^b	49.9^{bc}	5.5^d	56.3^c
		Max	101.3	86.7	298.1	72.6	176.2	75.1	139.2	79.8	6.8	64.5
		Min	48.4	50.6	125.8	38.8	64.7	34.7	29	26.6	4.2	48.8

Continued

Beds lined with laterite	Lat1-3	Aver	51.7^{b}	78.1^{a}	118.7^{b}	76.9^{a}	65.4^{b}	78^{d}	71.9^{b}	55.5^{d}	5.4^{ef}	58.4^{d}
		Max	62	91.9	208.1	89.7	105.4	90.4	143.4	79.9	6.2	65
		Min	24.4	68.9	69.6	52.3	38.7	58.2	34.6	30.4	4.4	49.3
	Lat3-5	Aver	73.7^{cd}	67.7^{b}	174.5^{c}	64.9^{b}	94.8^{c}	67.6^{e}	81.5^{b}	48^{bd}	5.7^{f}	55.1^{e}
		Max	92.3	90.6	267.5	79.7	137.5	80.3	153.9	77.9	6.8	62
		Min	48.3	53.4	90.4	36.4	59.3	39.2	35.9	22.7	4.8	51.4
	Lat5-8	Aver	86.3^{d}	61.2^{d}	267.7^{e}	46.1^{d}	151.3^{d}	48.2^{f}	92.6^{b}	38.3^{eb}	6.7^{g}	44.7^{f}
		Max	115	85.4	267.5	67.9	250.2	68.9	166.7	72.1	7.8	54.3
		Min	52.7	45.8	92.5	27.1	92.5	26.4	37.1	14.2	6.1	35.6

Values in the same column followed by the same superscript letter (*i.e.* a, b, c...) are not significantly different at $p < 0.05$. Sch1-3 Sch3-5 and Sch5-8 = shale-filled beds with grain sizes of 1 - 3, 3 - 5 and 5 - 8 mm respectively; Lat1-3, Lat3-5 and Lat5-8 = laterite-filled beds with grain sizes of 1 - 3, 3 - 5 and 5 - 8 mm respectively.

4. Discussion

This study investigated the influence of grain size (1 - 3, 3 - 5 and 5 - 8 mm) of shale and laterite on the functioning of constructed wetlands treating domestic wastewater. With regard to plant growth in the wetland beds, the results showed greater development of *Pennisetum purpureum* in the shale-filled beds than in the laterite beds. However, for both filtration materials (shale and laterite), the smaller the grain size, the greater the plant biomass produced, as well as the diameter of the plant stumps. This is explained by the fact that fine-grained materials tend to increase the residence time of water in the constructed wetland substrate [16]; this allows greater uptake of nutrients and water by the plants, thereby promoting much better plant growth. Furthermore, the greater plant biomass recorded in shale-filled beds (between 13.8 and 14.7 kg/m^2) compared to laterite beds (between 11.2 and 12.5 kg/m^2) is justified by the nature of shale to retain or absorb water in wetland beds, making water available to plants [23]. This observation is consistent with the volumes of water returned by the wetland beds, which appeared to be lower at the outlet of the shale-lined beds than those of the laterite beds. Studies such as that by Ama *et al.* [40] have also shown that the hydraulic conductivity of shale is lower than that of laterite, leading to a longer residence time in the former material and hence to a greater availability of water in the substrate that can be taken up by the plant to ensure its growth.

Monitoring of the physicochemical parameters of the effluent and filtrates during the treatment trial revealed lower pH values in the filtrates than in the raw water. This situation, as far as pH is concerned, is the result of the biodegradation of organic matter and/or the metabolism of plant nutrient assimilation [41] or the mineralogical composition of the geomaterials. The decrease in pH in filtrates from beds lined with shale and laterite reflects an acidification of the environment, which could be explained by the composition of these materials on the one hand, and by nitrification and oxidation of organic matter on the other. In addition, the decrease in pH in the filtrates could be explained by the neutralization of H^+ ions in the water by rock oxides [42]. According to Pambrun [43], ammonium adsorption tends to lower the pH. Furthermore, Li *et al.* [44] have shown that a decrease in pH leads to a higher COD removal efficiency. However, the pH values of filtrates discharged from shale and laterite lined beds comply with the wastewater discharge standards in Côte d'Ivoire ($5.5 \leq pH \leq 8.5$) [45]. Furthermore, the increase in dissolved oxygen in the different bed filtrates is due to the aeration of the raw water during its application to the wetland beds, especially since the wetlands tested in the present study are those with vertical flow, which provides a high oxygenation rate in addition to the oxygen released at the top of the plant roots [46] [47]. As for the electrical conductivity (EC), the values remain significantly lower in the bed filtrates compared to the raw effluent. This difference in EC could be explained by the retention of dissolved salts contained in the wastewater by the substrates of the beds lined with shale and laterite. According to Tchobanoglous *et al.* [48], filtration mechanisms, absorption, ion exchange, oxidation, neutralization, precipitation and complexation, contribute to the retention of dissolved salts in the reactor filter bed during the passage of wastewater.

The results of the concentration of total suspended solids (TSS) in the raw wastewater and in the bed filtrates showed a significant reduction in the filtrates, especially in the beds lined with small particle size material, *i.e.* beds lined with 1 - 3 mm shale (20.3 - 59.5 mg/L) and 1 - 3 mm laterite (25.4 - 62 mg/L). This is mainly due to the physical retention of particles in suspension in the pores of the filtration materials, which provide greater filtration potential for TSS in wetland substrates [49]-[51]. Wetland substrates act as a sieve or filter, retaining particles larger than the pores. They largely retain all the relatively coarse material at the surface of the beds and the fine material at depth, which is retained by clogging between the pores or by interception and attachment to the grains [16]. Furthermore, the shale-filled beds showed the best TSS retention performance compared to the laterite beds. This difference can be explained by the difference in hydraulic conductivity between these beds. However, only the TSS concentration of the bed lined with shale of grain size 1-3 (Sch1-3) met the applicable national standards (50 mg/L) set by [45]. In addition, the TSS removal efficiency of the 1 - 3 mm shale bed (80.2%) is higher than that obtained by Abdelhakeem *et al.* [50] (75%).

In terms of the removal of organic compounds such as BOD_5 and COD, the filtrates from the shale and laterite lined beds had significantly lower concentrations than those in the raw water. This significant reduction in organic matter in the filtrates from the shale and laterite lined beds is attributed to the good colonization of the substrates by the purifying microorganisms and the good oxygenation of the filtration mass [1] [52]. In fact, the resting phases of wetlands such as those developed in this study (vertically draining wetlands) favor the biological oxidation of the carbon load and the release of pores, which is necessary for the metabolism of bacteria during the biodegradation of organic matter [53]. However, considering the average concentrations of BOD_5 and COD discharged at the wetland outlet in the filtrates of the beds lined with shale and laterite, it appears that the beds lined with materials (shale and laterite) with grain sizes of 1 - 3 mm and 3 - 5 mm (*i.e.* 48, 3 - 94.8 mgO_2/L [BOD_5] and 92.4 - 174.5 mgO_2/L [COD]) that are lower than the two limits for BOD_5 (100 mgO_2/L) and COD (300 mg O2/L) specified in the regulations for wastewater discharge in Côte d'Ivoire [45]. In addition, the purification efficiencies recorded for beds lined with shale and laterite of grain sizes 1 - 3 mm and 3 - 5 mm (67.6-84.7% [BOD_5] and 64.9-83% [COD]) are higher than those of Coulibaly *et al.* [54] [55] (65% - 70% COD) and Masharqa *et al.* [56] (78% BOD_5).Therefore, in order to optimize the grain size of the geomaterials, a reasonable choice of size would be 3 - 5 mm, for both shale and laterite, provided that the quality of the filtrates obtained from the wetlands filled with the latter is assumed to be without major inconvenience to the receiving environment.

The reduction in NTK concentrations recorded in beds lined with shale and laterite can be explained by the ability of these geomaterials to adsorb nitrogen compounds, as mentioned by Rao and Batra [57]. Similarly, the harmonious plant development observed in the wetland beds during the experiment is clearly dependent on the plants taking up nutrients from the wastewater, including nitrogen compounds, to meet their nutritional needs [58] [59]. In addition, several studies have shown the significant involvement of purifying organisms in the degradation of wastewater pollutants, including nitrogen compounds [52] [60]. However, in the present study, a much higher density of microorganisms was favored by *Pennisetum purpureum*, as in the work of Zahui *et al.* [28], who obtained a higher total bacterial flora in wetlands planted with *P. purpureum* than those of plants such as *Andropogon gayanus*, *Chrysopogon zizanioides*, *Echinochloa pyramidalis* and *Tripsacum laxum*. Furthermore, the smaller the grain size of the filtration materials, the higher the purification efficiency of the NTK, as in the work of Christos and Vassilios [61], where higher nitrogen yields were obtained in wetlands with fine gravel than in those with coarse gravel. This result can be explained by the smaller pore sizes of fine geomaterials, which maximize the fraction retained by inter-pore clogging phenomena. In addition, the very rapid occupation of the pores by suspended particles during wetland operation favors a longer contact time between the wastewater and the purifying organisms, resulting in a more refined treatment in wetlands lined with fine materials compared to coarse materials. However, the higher NTK purification yields observed in shale-lined beds can be explained by their physicochemical nature, specific surface area [17] and grain size. In the presence of water, the shale becomes friable, breaking into numerous layers and transforming into a highly plastic or swelling clay [62]. By swelling, the clay exerts additional mechanical pressure on the structure of the rock, which can lead to varying degrees of cracking, creating space for greater absorption and development of plant roots. This root development would have been beneficial for shale-lined wetlands, particularly as roots promote significant secretion of root exudates, which serve as a complementary energy source for the activity of purifying microorganisms [63] [64]. However, only the concentration of NTK in the bed lined with 1 - 3 mm shale (46.48 mg/L) remains below the limits (50 mg/L) allowed by the regulations for wastewater discharges in Côte d'Ivoire [45]. In addition, the NTK purification efficiency obtained on the 1 - 3 mm shale bed (72.2%) was higher than those obtained by Hube *et al.* [65] (34% - 67%). The wetland filtrates had significantly lower concentrations of total phosphorus (TP) than the raw water. These results are mainly attributed to a process of adsorption and precipitation on the filtration material [66] [67], but also to microbial assimilation [68] [69] of phosphorus in the substrates of the different beds. In addition, the removal of phosphorus compounds is also related to the ability of these materials to release iron, which can react with PO_3^{4-} in wastewater. Because of their high porosity, shale and laterite allow water to pass through them, causing dissociation of iron oxides in solution in the form of Fe^{3+} and Fe^{2+} ions, which form Fe(OOH)-P complexes with phosphorus, resulting in hydroxylated iron phosphate by precipitation [70]. Furthermore, the difference in total phosphorus removal performance between shale-filled and laterite-filled beds could be explained by the rapid flow of effluent in laterite-filled beds, resulting in a reduction in the contact time required for the physico-chemical reactions of adsorption or precipitation in wetlands. The good removal rates obtained with filters lined with medium and fine gravel can be explained by their nature. If the filter media is fine, this means that the particles in the media form a very fine porous layer with a very large specific surface area. This facilitates the adsorption of phosphate ions. The finer the filter bed, the longer the residence time of the effluent in the bed. This gives microorganisms enough time to form a biofilm on which to bind phosphate ions, leading to their reduction in the filtrates [30]. The higher the specific surface area of the filter bed, the greater the adsorption and dissolution of phosphate ions [71]. However, the average concentrations of total phosphorus discharged at the outlet of wetlands lined with both materials (shale and laterite) [varying between 3.7 and 6.7 mg/L] are below the limit (15 mg/L) indicated in the regulations for wastewater discharges in Côte d'Ivoire [45]. In addition, we note that the purification efficiencies recorded on beds lined with shale and laterite of grain sizes 1 - 3 mm, 3 - 5 mm and 5 - 8 mm (44.7% - 72.4%) are higher than those of Abdelhakeem *et al.* [50], (22 and 56%). Therefore, in order to optimize the grain size of the geomaterials, the choice would be limited to 3 - 5 mm to also allow the treatment of organic matter (COD and BOD_5).

The good removal efficiencies obtained with beds packed with medium and fine substrates can be explained by their nature. A fine filter bed means that the particles form a very fine porous layer, with a very large total pore surface area. This facilitates the adsorption of pollutants by this layer, resulting in their retention on the surface of the geomaterials (shale and laterite).

5. Conclusions

This study investigated the influence of grain size (1 - 3 mm, 3 - 5 mm and 5 - 8 mm) of shale and laterite on the operation of constructed wetlands treating domestic wastewater. The use of these geomaterials as filter materials with different grain sizes resulted in good removal of total suspended solids (TSS), chemical oxygen demand (COD), biochemical oxygen demand (BOD5), total nitrogen Kjedahl (NTK) and total phosphorus (TP) from the wastewater. The plant species *P. purpureum* used as vegetation material is well adapted to the different artificial wetland beds, with higher plant biomass production and plant stump diameter in beds lined with shale and laterite with grain sizes of 1 - 3 mm.

The highest efficiencies in the removal of effluent pollutants were obtained in constructed wetlands lined with shale with a grain size of 1 - 3 mm (TSS [80.2%], COD [83%], BOD5 [84.7%], NTK [72.2%] and TP [72.4%]) and laterite with a grain size of 1 - 3 mm (TSS [78.1%], COD [77%], BOD5 [78%], NTK [55.5%] and TP [58.4%]). However, the choice of geomaterials grain size (3 - 5 mm) could be an advantage compared to the effort and resources required to purchase a quantity of geomaterials to obtain a grain size of 1 - 3 mm. This is because the filtrates discharged from beds lined with geomaterials (shale and laterite) are expected to be free of major organic and phosphorus contaminants. However, increasing the depth of the wetlands to be lined with 3 - 5 mm shale and laterite beyond that of the present study would refine the treatment of nitrogen compounds and total suspended solids.

Conflicts of Interest

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

References

[1] Hassan, I., Chowdhury, S.R., Prihartato, P.K. and Razzak, S.A. (2021) Wastewater Treatment Using Constructed Wetland: Current Trends and Future Potential. *Processes*, **9**, Article 1917. https://doi.org/10.3390/pr9111917

[2] Vymazal, J. (2022) The Historical Development of Constructed Wetlands for Wastewater Treatment. *Land*, **11**, Article 174. https://doi.org/10.3390/land11020174

[3] de Campos, S.X. and Soto, M. (2024) The Use of Constructed Wetlands to Treat Effluents for Water Reuse. *Environments*, **11**, Article 35. https://doi.org/10.3390/environments11020035

[4] Waly, M.M., Ahmed, T., Abunada, Z., Mickovski, S.B. and Thomson, C. (2022) Constructed Wetland for Sustainable and Low-Cost Wastewater Treatment: Review Article. *Land*, **11**, Article 1388. https://doi.org/10.3390/land11091388

[5] Chen, J., Guo, F., Wu, F. and Bryan, B.A. (2023) Costs and Benefits of Constructed Wetlands for Meeting New Water Quality Standards from China's Wastewater Treatment Plants. *Resources*, *Conservation and Recycling*, **199**, Article ID: 107248. https://doi.org/10.1016/j.resconrec.2023.107248

[6] Avellán, T. and Gremillion, P. (2019) Constructed Wetlands for Resource Recovery in Developing Countries. *Renewable and Sustainable Energy Reviews*, **99**, 42-57. https://doi.org/10.1016/j.rser.2018.09.024

[7] Stefanakis, A. (2019) The Role of Constructed Wetlands as Green Infrastructure for Sustainable Urban Water Management. *Sustainability*, **11**, Article 6981. https://doi.org/10.3390/su11246981

[8] Agaton, C.B. and Guila, P.M.C. (2023) Ecosystem Services Valuation of Constructed Wetland as a Nature-Based Solution to Wastewater Treatment. *Earth*, **4**, 78-92. https://doi.org/10.3390/earth4010006

[9] Matter, N.M. and Gado, N.G. (2024) Constructed Wetlands Nature-Based Solutions to Enhance Urban Resilience in Egyptian Cities. *HBRC Journal*, **20**, 231-255. https://doi.org/10.1080/16874048.2024.2311521

[10] Rahman, M.E., Bin Halmi, M.*I.E*., Bin Abd Samad, M.Y., Uddin, M.K., Mahmud, K., Abd Shukor, M.Y., *et al.* (2020) Design, Operation and Optimization of Constructed Wetland for Removal of Pollutant. *International Journal of Environmental Research and Public Health*, **17**, Article 8339. https://doi.org/10.3390/ijerph17228339

[11] Kulshreshtha, N.M., Verma, V., Soti, A., Brighu, U. and Gupta, A.B. (2022) Exploring the Contribution of Plant Species in the Performance of Constructed Wetlands for Domestic Wastewater Treatment. *Bioresource Technology Reports*, **18**, Article ID: 101038. https://doi.org/10.1016/j.biteb.2022.101038

[12] Coundoul, F., Ndiaye, A.K., Deme, A. and Armegnol, A.T. (2024) A Comparative Analysis of Vetiver and *Typha* in Ecological Wastewater Treatment Using a Horizontal Flow Constructed Wetlands in Rural Setting. *Journal of Agricultural Chemistry and Environment*, **13**, 67-82. https://doi.org/10.4236/jacen.2024.131005

[13] Sanjrani, M.A., Zhou, B., Zhao, H., Zheng, Y.P., Wang, Y. and Xia, S.B. (2019) The Influence of Wetland Media in Improving the Performance of Pollutant Removal During Water Treatment: A Review. *Applied Ecology and Environmental Research*, **17**, 3803-3818. https://doi.org/10.15666/aeer/1702_38033818

[14] Ji, Z., Tang, W. and Pei, Y. (2022) Constructed Wetland Substrates: A Review on Development, Function Mechanisms, and Application in Contaminants Removal. *Chemosphere*, **286**, 131564. https://doi.org/10.1016/j.chemosphere.2021.131564

[15] Sandoval Herazo, L.C., Alvardo-Lassman, A., Marín-Muñiz, J.L., Rodríguez-Miranda, J.P. and Fernández-Lambert, G. (2023) A Critical Review of Mineral Substrates Used as Filter Media in Subsurface Constructed Wetlands: Costs as a Selection Criterion. *Environmental Technology Reviews*, **12**, 251-271. https://doi.org/10.1080/21622515.2023.2198146

[16] Wang, L., Ma, L., Wang, J., Zhao, X., Jing, Y., Liu, C., *et al.* (2024) Research Progress on the Removal of Contaminants from Wastewater by Constructed Wetland Substrate: A Review. *Water*, **16**, Article 1848. https://doi.org/10.3390/w16131848

[17] Koua-Koffi, N.A.A., Coulibaly, L.S., Sangare, D. and Coulibaly, L. (2018) Laterite, Sandstone and Shale as Adsorbents for the Removal of Arsenic from Water. *American Journal of Analytical Chemistry*, **9**, 340-352. https://doi.org/10.4236/ajac.2018.97027

[18] Koua-Koffi, N.A.A., Coulibaly, L.S., Sangare, D. and Coulibaly, L. (2021) Removal of Arsenic from Simulated Groundwater Using Calcined Shale as the Adsorbent. *International Research Journal of Pure and Applied Chemistry*, **22**, 31-39. https://doi.org/10.9734/irjpac/2021/v22i630414

[19] Miranda, S.T., Matos, A.T.D., Matos, M.P.D., Saraiva, C.B. and Teixeira, D.L. (2019) Influence of the Substrate Type and Position of Plant Species on Clogging and the Hydrodynamics of Constructed Wetland Systems. *Journal of Water Process Engineering*, **31**, Article ID: 100871. https://doi.org/10.1016/j.jwpe.2019.100871

[20] Witthayaphirom, C., Chiemchaisri, C. and Chiemchaisri, W. (2019) Optimization of

Reactive Media for Removing Organic Micro-Pollutants in Constructed Wetland Treating Municipal Landfill Leachate. *Environmental Science and Pollution Research*, **27**, 24627-24638. https://doi.org/10.1007/s11356-019-06010-3

[21] Zhao, L.L., Shao, X.X., Wu, M., Jiang, X.S. and Lu, L.Y. (2018) Effects of Different Substrates and Particle Sizes on Wastewater Purification. *Environmental Science*, **39**, 4236-4241. https://doi.org/10.13227/j.hjkx.201712009

[22] Song, H., Feng, J., Zhang, L., Yin, H., Pan, L., Li, L., *et al.* (2021) Advanced Treatment of Low C/N Ratio Wastewater Treatment Plant Effluent Using a Denitrification Biological Filter: Insight into the Effect of Medium Particle Size and Hydraulic Retention Time. *Environmental Technology & Innovation*, **24**, Article ID: 102044. https://doi.org/10.1016/j.eti.2021.102044

[23] Fatim Traoré, N., Pétémanagnan Ouattara, J., Michaël Zahui, F., Cyrille Beda, A.J. and Messou, A. (2024) Assessment of Secondary Metabolites in *Pennisetum Purpureum* Planted into Constructed Wetlands Using Shale and Laterite as Substrate for Wastewater Treatment. *Heliyon*, **10**, e33284. https://doi.org/10.1016/j.heliyon.2024.e33284

[24] Cominato, V., Benavente Canteras, F., Andréia Gachet, L. and Cristina Cecche Lintz, R. (2022) The Effect of Granulometry of Natural and Recycled Coarse Aggregate on Permeable Concrete Properties. *Materials Today: Proceedings*, **65**, 1711-1718. https://doi.org/10.1016/j.matpr.2022.04.717

[25] Pucher, B. and Langergraber, G. (2019) The State of the Art of Clogging in Vertical Flow Wetlands. *Water*, **11**, Article 2400. https://doi.org/10.3390/w11112400

[26] Ergaieg, K., Msaddek, M.H., Kallel, A. and Trabelsi, I. (2021) Monitoring of Horizontal Subsurface Flow Constructed Wetlands for Tertiary Treatment of Municipal Wastewater. *Arabian Journal of Geosciences*, **14**, Article No. 2045. https://doi.org/10.1007/s12517-021-08419-y

[27] Kouassi, A.M., Kouao, J. and Kouakou, K.E. (2022) Caractérisation intra-annuelle de la variabilité climatique en Côte d'Ivoire. *Bulletin de l'Association de géographes français*, **99**, 289-306. https://doi.org/10.4000/bagf.9534

[28] Zahui, F.M., Ouattara, J.P., Kamagaté, M., Coulibaly, L. and Stefanakis, A.I. (2021) Effect of Plant Species on the Performance and Bacteria Density Profile in Vertical Flow Constructed Wetlands for Domestic Wastewater Treatment in a Tropical Climate. *Water*, **13**, Article 3485. https://doi.org/10.3390/w13243485

[29] Rooklidge, S.J. and Ketchum, L.H. (2002) Calcite-amended Horizontal Roughing Filtration for Clay Turbidity Removal. *Journal of Water Supply: Research and Technology-Aqua*, **51**, 333-342. https://doi.org/10.2166/aqua.2002.0030

[30] Ama-Cauphys, A.B., Akpo, S.K., Coulibaly, S.L., Ouattara, P.J. and Coulibaly, L. (2016) Performance of Rock Materials (Shale, Laterite and Granite) towards the Removal of Orthophosphates from Wastewater. *International Journal of Innovation and Scientific Research*, **21**, 402-413.

[31] ISO (International Standardization Organization) (2008) 10523: Water Quality-Determination of pH-Analytical Measurement. 2nd Edition, ISO.

[32] ISO (International Standardization Organization) (2012) 5814: Water Quality-Determination of Dissolved Oxygen-Electrochemical Probe Method. 3rd Edition, ISO.

[33] ISO (International Standardization Organization) (1985) 7888: Water Quality-Determination of Conductivité—Analytical Measurement. ISO.

[34] ISO (International Standardization Organization) (1997) 11923: Water Quality-Determination of Suspended Solids by Filtration through Glassfibre Filters. ISO.

[35] ISO (International Standardization Organization) (1989) 6060/2: Water Quality-

Determination of Chemical Oxygen Demand-Potassium Dichromate Method. 2nd Edition, ISO.

[36] ISO (International Standardization Organization) (2003) 5815/1: Water Quality-Determination of Biochemical Oxygen Demand after n Days (BODn)-Part 1: Dilution and Seeding Method with Allylthiourea Addition. ISO.

[37] ISO (International Standardization Organization) (1984) 5663: Water Quality-Determination of Kjedahl Nitrogen-Method after Mineralization with Selenium. ISO.

[38] ISO (International Standardization Organization) (2004) 6878: Water Quality-Determination of Phosphorus-Ammonium Molybdate Spectrometric Method. 2nd Edition, ISO.

[39] Abissy, M. and Mandi, L. (2005) Utilisation des plantes aquatiques enracinées pour le traitement des eaux usées urbaines: Cas du roseau. *Revue des sciences de l'eau*, **12**, 285-315. https://doi.org/10.7202/705353ar

[40] Ama-Cauphys, B.A., Kouamé, A.K., Messou, A. and Coulibaly, L. (2022) Influence de la hauteur du massif filtrant sur l'élimination des polluants organiques dans un filtre à sable à alimentation intermittente. *International Journal of Biological and Chemical Sciences*, **15**, 2233-2246. https://doi.org/10.4314/ijbcs.v15i5.41

[41] Kone, M., Zongo, I., Bonou, L., Koulidiati, J., Joly, P., Bouvet, Y., *et al.* (2011) Traitement d'eaux résiduaires urbaines par filtres plantés à flux vertical sous climat Soudano-Sahélien. *International Journal of Biological and Chemical Sciences*, **5**, 217-231. https://doi.org/10.4314/ijbcs.v5i1.68100

[42] De Haas, D., Wentzel, M. and Ekama, G. (2004) The Use of Simultaneous Chemical Precipitation in Modified Activated Sludge Systems Exhibiting Biological Excess Phosphate Removal: Part 6: Modelling of Simultaneous Chemical-Biological P Removal—Review of Existing Models. *Water SA*, **27**, 135-150. https://doi.org/10.4314/wsa.v27i2.4988

[43] Pambrun, V. (2005) Analyse et modélisation de la nitrification partielle et de la précipitation concomitante du phosphore dans un réacteur à alimentation séquencée. Master's Thesis, Institut National des Sciences Appliquées de Toulouse.

[44] Li, Q., Ma, Y., Chen, J., Cai, Y., Lu, Q., Yu, Q., *et al.* (2023) The Negative Effect of the High pH of Waste Concrete in Constructed Wetlands on COD and N Removal. *Journal of Water Process Engineering*, **51**, Article ID: 103356. https://doi.org/10.1016/j.jwpe.2022.103356

[45] Ministère de l'environnement, des eaux et forêts (2008) Arrêté N°01164/MINEEF/CIAPOL/SDIIC du 04 Nov. 2008, portant Règlementation des Rejets et Emissions des Installations Classées pour la Protection de l'Environnement. Ministère de l'Environnement, des Eaux et Forêts/Centre Ivoirien Antipollution, 20.

[46] Nivala, J., Wallace, S., Headley, T., Kassa, K., Brix, H., van Afferden, M., *et al.* (2013) Oxygen Transfer and Consumption in Subsurface Flow Treatment Wetlands. *Ecological Engineering*, **61**, 544-554. https://doi.org/10.1016/j.ecoleng.2012.08.028

[47] Pérez, M.M., Hernández, J.M., Bossens, J., Jiménez, T., Rosa, E. and Tack, F. (2014) Vertical Flow Constructed Wetlands: Kinetics of Nutrient and Organic Matter Removal. *Water Science and Technology*, **70**, 76-81. https://doi.org/10.2166/wst.2014.183

[48] Tchobanoglous, G., Burton, F.L. and Stensel, H.D. (2003) Wastewater Engineering: Treatment Disposal and Reuse. 4th Edition, McGraw-Hill, 1334.

[49] Ouattara, P.J.M., Coulibaly, L., Manizan, P. and Gourène, G. (2008) Traitement des eaux résiduaires urbaines par un marais artificiel à drainage vertical planté avec Panicum maximum sous climat tropical. *European Journal of Scientific Research*, **23**, 25-

40.

[50] Abdelhakeem, S.G., Aboulroos, S.A. and Kamel, M.M. (2016) Performance of a Vertical Subsurface Flow Constructed Wetland under Different Operational Conditions. *Journal of Advanced Research*, **7**, 803-814. https://doi.org/10.1016/j.jare.2015.12.002

[51] Bohórquez, E., Paredes, D. and Arias, C.A. (2016) Vertical Flow-Constructed Wetlands for Domestic Wastewater Treatment under Tropical Conditions: Effect of Different Design and Operational Parameters. *Environmental Technology*, **38**, 199-208. https://doi.org/10.1080/09593330.2016.1230650

[52] Wang, J., Long, Y., Yu, G., Wang, G., Zhou, Z., Li, P., *et al.* (2022) A Review on Microorganisms in Constructed Wetlands for Typical Pollutant Removal: Species, Function, and Diversity. *Frontiers in Microbiology*, **13**, Article 845725. https://doi.org/10.3389/fmicb.2022.845725

[53] Molle, P., Liénard, A., Boutin, C., Merlin, G. and Iwema, A. (2005) How to Treat Raw Sewage with Constructed Wetlands: An Overview of the French Systems. *Water Science and Technology*, **51**, 11-21. https://doi.org/10.2166/wst.2005.0277

[54] Coulibaly, L., Kouakou, J., Savané, I. and Gourène, G. (2008) Domestic Wastewater Treatment with a Vertical Completely Drained Pilot Scale Constructed Wetland Planted with Amaranthus Hybridus. *African Journal of Biotechnology*, **7**, 2656-2664.

[55] Coulibaly, L., Savane, I. and Gourene, G. (2008) Domestic Wastewater Treatment with a Vertical Completely Drained Pilot Scale Constructed Wetland Planted with Corchorus Olitérius. *African Journal of Agricultural Research*, **3**, 587-596.

[56] Masharqa, A., Al-Tardeh, S., Mlih, R. and Bol, R. (2023) Vertical and Hybrid Constructed Wetlands as a Sustainable Technique to Improve Domestic Wastewater Quality. *Water*, **15**, Article 3348. https://doi.org/10.3390/w15193348

[57] Rao, D.L.N. and Batra, L. (1983) Ammonia Volatilization from Applied Nitrogen in Alkali Soils. *Plant and Soil*, **70**, 219-228. https://doi.org/10.1007/bf02374782

[58] Abbasi, H., Vasileva, V. and Lu, X. (2017) The Influence of the Ratio of Nitrate to Ammonium Nitrogen on Nitrogen Removal in the Economical Growth of Vegetation in Hybrid Constructed Wetlands. *Environments*, **4**, Article 24. https://doi.org/10.3390/environments4010024

[59] Tho, B.T., Lambertini, C., Eller, F., Brix, H. and Sorrell, B.K. (2017) Ammonium and Nitrate Are Both Suitable Inorganic Nitrogen Forms for the Highly Productive Wetland Grass Arundo Donax, a Candidate Species for Wetland Paludiculture. *Ecological Engineering*, **105**, 379-386. https://doi.org/10.1016/j.ecoleng.2017.04.054

[60] Rani, A., Chauhan, M., Kumar Sharma, P., Kumari, M., Mitra, D. and Joshi, S. (2024) Microbiological Dimensions and Functions in Constructed Wetlands: A Review. *Current Research in Microbial Sciences*, **7**, Article ID: 100311. https://doi.org/10.1016/j.crmicr.2024.100311

[61] Akratos, C.S. and Tsihrintzis, V.A. (2007) Effect of Temperature, HRT, Vegetation and Porous Media on Removal Efficiency of Pilot-Scale Horizontal Subsurface Flow Constructed Wetlands. *Ecological Engineering*, **29**, 173-191. https://doi.org/10.1016/j.ecoleng.2006.06.013

[62] Okeke, C. and Okogbue, O.C. (2011) Shales: A Review of Their Classifications, Properties and Importance to the Petroleum Industry. *Global Journal of Geological Sciences*, **9**, 55-73.

[63] Zhai, X., Piwpuan, N., Arias, C.A., Headley, T. and Brix, H. (2013) Can Root Exudates from Emergent Wetland Plants Fuel Denitrification in Subsurface Flow Constructed Wetland Systems? *Ecological Engineering*, **61**, 555-563. https://doi.org/10.1016/j.ecoleng.2013.02.014

[64] Weber, K.P. and Gagnon, V. (2014) Microbiology in Treatment Wetlands. *Sustainable Sanitation*, **18**, 25-30.

[65] Hube, S., Zaqout, T., Ögmundarson, Ó., Andradóttir, H.Ó. and Wu, B. (2023) Constructed Wetlands with Recycled Concrete for Wastewater Treatment in Cold Climate: Performance and Life Cycle Assessment. *Science of the Total Environment*, **904**, Article ID: 166778. https://doi.org/10.1016/j.scitotenv.2023.166778

[66] Molle, P., Martin, S., Esser, D., Besnault, S., Morlay, C. and Harouiya, N. (2011) Phosphorus Removal by the Use of Apatite in Constructed Wetlands: Design Recommendations. *Water Practice and Technology*, **6**, 1629-1637. https://doi.org/10.2166/wpt.2011.046

[67] Vohla, C., Kõiv, M., Bavor, H.J., Chazarenc, F. and Mander, Ü. (2011) Filter Materials for Phosphorus Removal from Wastewater in Treatment Wetlands—A Review. *Ecological Engineering*, **37**, 70-89. https://doi.org/10.1016/j.ecoleng.2009.08.003

[68] Nguyen, H.T.T., Le, V.Q., Hansen, A.A., Nielsen, J.L. and Nielsen, P.H. (2011) High Diversity and Abundance of Putative Polyphosphate-Accumulating Tetrasphaera-Related Bacteria in Activated Sludge Systems. *FEMS Microbiology Ecology*, **76**, 256-267. https://doi.org/10.1111/j.1574-6941.2011.01049.x

[69] Stokholm-Bjerregaard, M., McIlroy, S.J., Nierychlo, M., Karst, S.M., Albertsen, M. and Nielsen, P.H. (2017) A Critical Assessment of the Microorganisms Proposed to Be Important to Enhanced Biological Phosphorus Removal in Full-Scale Wastewater Treatment Systems. *Frontiers in Microbiology*, **8**, Article 718. https://doi.org/10.3389/fmicb.2017.00718

[70] Zak, D. and Gelbrecht, J. (2002) Phosphorus Retention at the Redox Interface of Peat Lands Adjacent to Surface Waters. *Wasser & Boden*, **54**, 71-76.

[71] Garzón-Zúñiga, M.A., Navarro-Franco, J.A. and Moreno Andrade, I. (2024) Filter Packed with Al-Sludge Waste for Phosphorus Removal as a Polishing System in a Wastewater Treatment Plant. *Tecnología y ciencias del agua*, **15**, 311-353. https://doi.org/10.24850/j-tyca-2024-06-07

Appendix

Figure A1. Illustration of the crushing and screening stage of the geomaterials.

Figure A2. Overview of the experimental plant after one month of operation.