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A Comparative Performance Assessment of the Integrated Upflow and Surface Flow-Based Constructed Wetlands Dosed with Landfill Leachate: Electrode Coupling and Input Load Variation

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Abstract: This study reports organic, nutrient, and coliform removal performances of two integrated wetlands designed to treat landfill leachate. Each integrated system included two components: a normal or electrode-integrated upflow-based wetland and a surface flow wetland (with internal baffle walls). The components were fully or partially filled with stone dust media and planted with Canna indica. Two hydraulic loading rates, i.e., 15 L and 60 L (per day), were applied. The integrated wetlands achieved a mean biochemical oxygen demand (BOD), chemical oxygen demand (COD), total nitrogen (TN), total phosphorus (TP), and coliform removal efficiency ranges of 89–94%, 95–97%, 85–91%, 91–98%, and 70–88%, respectively, within the applied loading ranges. The electrode-dependent system achieved better pollutant removal performances due to the influence of electrochemical-based bioreactions that fostered microbial decomposition. Nitrogen accumulation percentage (with respect to observed removal) in plant tissues ranged between 0.6 and 25%; phosphorus accumulation percentage was negligible, i.e., $\leq$0.009%. The chemical composition of the stone dust media supported nutrient adsorption. Stable nutrient removal performance was observed with both systems despite variable loading ranges due to pollutant removal in the upflow-based wetlands followed by controlled flow direction (induced by baffle walls) in the surface flow wetlands that triggered chemical and biological removals. Mean power density production ranged between 235 and 946 mW/m^3 with the electrode-based integrated wetland system. In summary, this study demonstrates the application of integrated wetland systems to treat landfill leachate and the associated factors to achieve stable removal under variable loading ranges.

Keywords: adsorption; baffle wall; electrochemical oxidation; organic strength; performance stability; plant uptake

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1. Introduction

Solid wastes generated from urban areas are often compiled in landfill sites because such a process is inexpensive [1,2]. Rainfall, microbial decomposition, and moisture content release produce leachate wastewater (from the compiled wastes of landfill sites) with substantial pollutant composition [3–5]. Hence, wastewater treatment systems are required in landfill sites to remove the leachate pollutant and maintain equilibrium in the surrounding environment [1,6,7]. Because of the complex composition, chemical (precipitation, advanced oxidation, and coagulation–flocculation), physical–chemical (adsorption, membrane, and air strip), electrochemical-based (oxidation, reduction, coagulation, Fenton, three-dimensional electrodes, and ion exchange membrane), and biological (aerobic and anaerobic) treatment systems have been employed for leachate wastewater treatment [5,6,8–10]. Although chemical-based technologies are highly efficient for landfill leachate treatment, excessive chemical requirements and substantial sludge generation of such technologies increase operational costs that could be counterbalanced by biological methods due to their cost-effective characteristics [5]. However, these systems often exhibit inefficiencies in removing refractory compounds from leachate wastewater [9,10].

Constructed wetlands, plants, and media-packed natural-based biological systems that utilize their components to support the growth of microbial flora have been implemented for landfill leachate treatment due to possessing some unique advantages such as low cost, easy operation, and functioning capacities in centralized or decentralized areas [8,11–13]. A literature review indicates a mean of 21–97% organic, 16–99% nitrogen, and 61–100%, phosphorus removals with constructed wetlands employed worldwide for landfill leachate treatment [8,14–22]. Wider reported removal efficiency deviations could be linked to the variations in operational parameters (effluent recirculation, leachate co-treatment, wetland aeration, and stages), wetland components (media and plant), and climatic conditions. The operational parameters appeared to be the critical performance optimizing factors (in the reported studies) due to their capacity to alter the leachate's chemical composition or create environmental conditions inside the media that favored different chemical and microbial-based pollutant removal routes [17,19,22].

The positive impact of the operational factors (effluent recirculation, leachate co-treatment, and wetland aeration) on the pollutant removal performance of the leachate treatment-based wetlands is also accompanied by some disadvantages, such as operational cost increments due to pump requirements and fossil fuel consumption. Regarding the remaining operational factor, i.e., wetland stages, a review article by Bakhshoodeh, Alavi [8] reported better performance of hybrid stage-based wetlands (combination between any two or three wetland types: vertical, horizontal, and surface flow) employed worldwide for landfill leachate treatment. Additional contact between wetland components and wastewater pollutants, along with exposure to alternative aerobic–anoxic/anaerobic environments (inside the media), might have improved the operational performances of the reviewed hybrid wetland systems. However, such a hybrid setup demands substantial land requirements that might increase overall construction costs, particularly in areas where land availability is a constraint.

Integrated wetlands that include special internal structural configurations to control flow directions inside the media have been reported for different types of wastewater treatment [23–29]. Due to controlled vertical and horizontal flow directions (induced by the internal structural arrangement), these wetlands could maximize wastewater contact with the wetland components and environmental pockets (inside the bed matrix) in compacted areas [30]. Efficient treatment of the integrated wetlands despite being dosed with high organic-strength wastewater [31] suggests their potential application for raw landfill leachate treatment, particularly under financial constraints or limited land availability, but this has not been reported.

Electrode-integrated wetlands that rely on electrochemical bioreactions to achieve wastewater pollutant reduction and concurrent bioenergy production [32–34] have also been investigated recently to treat landfill leachate [11,35,36]. Such reported leachate treatment-based experimental setups included electrode-integrated single-stage (tidal flow-based) or hybrid (vertical followed by horizontal flow) wetland trains. These studies reported better pollutant removal performance of the electrode-integrated hybrid systems than the single stages, even though the hybrid systems received raw leachate and the single stages were dosed with a mixture of sewage and leachate wastewater (i.e., co-treatment). Moreover, the electrode-integrated hybrid wetland arrangements achieved better pollutant removal (from leachate wastewater) than parallelly operated normal (without electrode integration) hybrid systems due to the additional influence of electrochemical-based reactions. Such advantages of the bioenergy-producing wetlands could be extended to the integrated wetlands to improve their operational performance, particularly when employed to treat raw landfill leachate. The notable pollutant removal of electrode-based and integrated upflow–downflow-based wetlands designed by Liu, Sun [37], despite receiving high organic-strength swine wastewater, also supports their probable application to treat landfill leachate. A comparative experimental trial design and execution with normal and bioenergy-producing integrated wetlands for landfill leachate treatment would provide more accurate information on the associated pollutant removal pathways and the influencing factors but has not been reported to date.

This study abridged some knowledge gaps associated with the application of normal and electrode-based integrated wetlands to treat landfill leachate. Two integrated wetland systems were designed; each system included two compartments: an upflow-based wetland followed by a surface flow wetland (with internal baffle walls to control flow directions). Of the two systems, one operated as a normal (without electrode integration) unit; the remaining system operated as an electrode-dependent unit. Such a comparative experimental protocol was designed to assess the impact of electrodes and associated bioreactions on pollutant removal from landfill leachate wastewater. The developed systems were operated under variable hydraulic loading rates to investigate the dependency between electrochemically active–inactive pollutant removal routes and input loading variations. As such, the main objective of this research was to assess the synergistic impact of controlled flow direction, common (plant, media), and additional (i.e., electrodes) wetland components on pollutant (organic, nutrient, and coliform) removal performances with integrated wetland systems under variable leachate wastewater loading ranges.

2. Materials and Methods

2.1. Construction of the Wetlands

Figure 1 depicts a detailed diagram of the experimental arrangement that included two rectangular tanks (i.e., systems 1 and 2) constructed with steel plates. The length, width, and height of each tank were 1.6 m, 0.8 m, and 0.45 m, respectively. Each of the two systems was divided into two compartments by a solid steel bar 0.3 m high while maintaining a horizontal distance of 0.4 m or 1.2 m from the tank's left- or right-side wall, respectively. Two baffle walls were integrated into the second compartment of each system. The height of the first baffle wall was 0.3 m and was positioned at a horizontal distance of 0.8 m from the left- or right-side wall of each system. The height of the second baffle wall was 0.25 m and maintained a horizontal distance of 1.2 m or 0.4 m from the left- or right-side wall of each system. A clear spacing of 0.2 m was provided between the bottom portions of the second baffle wall and the rectangular tank (of each system). Therefore, these baffle walls divided the second compartment into three segments/chambers.

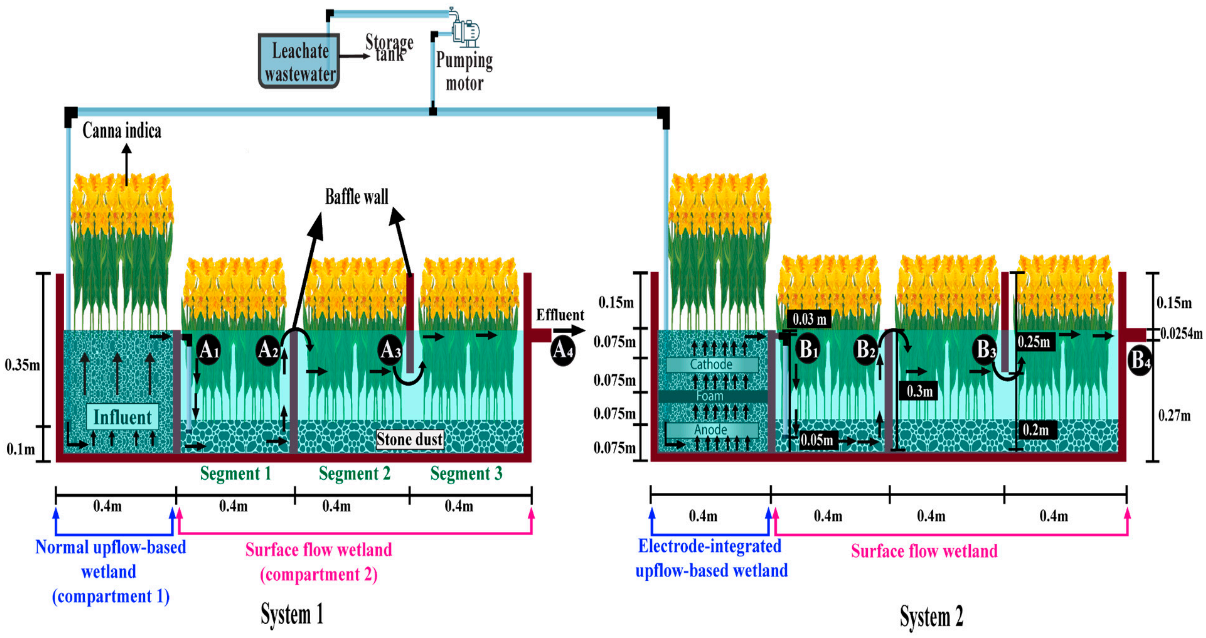

Figure 1. Integrated normal (system 1) and electrode-dependent (system 2) constructed wetlands for landfill leachate treatment.

Stone dust materials (size: 4–10 mm; porosity: 28%) were selected to fill both compartments (in systems 1 and 2) wholly or partially. The first compartment of the two systems was completely filled with stone dust materials that achieved a depth of 0.3 m. The second compartment of both systems was partially filled with stone dust, achieving a depth of 0.1 m. Two steel-built anode and cathode electrodes were buried inside the stone dust media of the first compartment in system 2. The length and width of a single anode or cathode electrode were 0.3 and 0.5 m, respectively. The anode and cathode electrodes were positioned 0.15 m apart (inside the media) and were separated by a glass wool thermal foam. The distance of the buried cathode or anode electrode from the top or bottom portion of the media was 0.075 m. The electrodes were connected by copper wires, an 820 Ω resistor, and a multimeter. As the electrode was the only variable within the two systems, they are referred to as system 1 (normal/without electrode) and system 2 (electrode-dependent).

Canna indica was selected as the wetland plant in this study; such species produce higher biomass, which favors the development of a diverse microbial population [38]. The selected species were planted into the stone dust media of the two systems. After plantation, the two systems were waterlogged for 12 weeks to allow plant growth and maturation.

2.2. Landfill Leachate Properties

Leachate wastewater was collected from the Aminbazar landfill site, which Dhaka North City Corporation (Dhaka, Bangladesh) manages. The collected leachate was stored in a water-holding tank for dosing. The mean pollutant composition of the collected landfill leachate is summarized in Table 1. The mean pH concentration indicates the alkaline nature of the landfill leachate, whereas the redox (E_h) profiles signify its anoxic nature. The influent landfill leachate comprised inorganic nitrogen (NH_4-N, NO_2-N, and NO_3-N) and organic nitrogen (due to deviation between TN and the summation of inorganic nitrogen components). NO_3-N was the major nitrogen component, contributing to 68% of the overall TN concentration; NH_4-N contributed to 18% of the overall TN concentration. Organic nitrogen primarily contributed to the remaining proportion, as NO_2-N concentration in the landfill leachate was very low. The leachate's mean organic biodegradation ratio (BOD/COD) was 0.3, indicating the limited presence of biodegradable organic compounds. A very low biodegradation ratio of the collected leachate wastewater (from the Aminbazar landfill site) was also reported previously due to its medium age and unstable conditions [39].

Table 1. Mean composition of the landfill leachate. Standard deviation values are presented within the brackets.

Parameters	Units	Concentration
pH	-	8.7 (0.4)
E_h	mV	10.6 (110.8)
NH_4-N	mg/L	33.4 (7.9)
NO_2-N	mg/L	0.9 (1.0)
NO_3-N	mg/L	127.8 (77.6)
TN	mg/L	188.7 (82.5)
TP	mg/L	208.3 (108.0)
BOD	mg/L	368.8 (95.0)
COD	mg/L	1217.1 (422.1)
Coliform	CFU/100 mL	23,571 (2158.0)

2.3. Wastewater Dosing

The two systems received raw leachate wastewater for 34 weeks. The first 13 weeks (of hydraulic dosing) allowed systems adaptation. Experimental runs were conducted within the latter 21 weeks. The overall experimental protocol was divided into two phases: I and II. In Phase I, each of the two systems received 15 L of leachate wastewater (per day) for 12 weeks. The hydraulic load was increased four-fold, i.e., 60 L across each system (per day) during Phase II, which was continued for 9 weeks. Both systems received wastewater continuously (i.e., 24 h) during the two operational phases. The experimental protocol did not include a leachate pretreatment step, as this research was designed to assess the capacity of integrated constructed wetlands (as the sole technology) in polishing landfill leachate.

Leachate wastewater was pumped from the holding tank to the bottom portion of the first compartment (in system 1), which was filled with stone dust media. The pumped wastewater flowed from the bottom to the top media portion in the first compartment in system 1. Therefore, the first compartment in system 1 operated as an upflow-based normal vertical flow wetland. The wastewater dosing protocol with system 2 was similar to the other system. Leachate wastewater was pumped from the holding tank to the bottom portion of the first compartment and was forced to pass the anode (bottom media portion) and cathode (top media portion) zones.

The effluent of each system's upflow-based wetland (first compartment) was transferred into the subsequent second compartment through an effluent collection valve located at a vertical depth of 0.27 m (measured from the bottom of the tank). Such an effluent collection valve was connected with a rubber pipe that was buried at a 0.05 m depth inside the media (measured from the bottom of the tank) of the first segment/chamber in the second compartment. The effluent flowed through the connected rubber pipe (under gravity) inside the media of the first segment (in the second compartment), then flowed upward, and overflowed through the first baffle wall to the following segment in the second compartment. Such an arrangement prevented flow short circuiting from the first to the second segment in the second compartment. The flow was further intercepted in a downward–upward direction by the second baffle wall while flowing from the second to the third segment in the second compartment. The effluent from the third segment (that was also the effluent of the second compartment and the whole system) was collected by an effluent collection valve located at a 0.27 m depth measured from the bottom of the tank. Because of maintaining a free water surface, the second compartments of both systems operated as surface flow wetlands. In this study, systems 1 and 2 have been referred to as integrated normal (without electrode) and electrode-dependent wetlands, respectively, due to the combination of vertically upward–downward and surface flow regimes in a single unit.

2.4. Sample Collection and Analysis Protocols

The samples were taken weekly from the leachate holding tank and the outlet of the normal (sample collection point A_1 in system 1—Figure 1) and electrode-integrated upflow-based wetlands (sample collection point B_1 in system 2—Figure 1). The samples were also extracted from the first segment (sample collection points A_2 in system 1 and B_2 in system 2) and the second segment (sample collection points A_3 in system 1 and B_3 in system 2) of the surface flow wetlands. The effluent of the third segment of the surface flow wetlands (also the final effluent of the surface flow wetlands or both systems) was collected from the outlets (sample collection points A_4 in system 1 and B_4 in system 2). This sampling protocol allowed us to assess the removal performance of the upflow-based wetlands and the segments of the surface flow wetlands, along with the overall performance of both systems.

The pH and E_h concentration of the influent and effluent samples were measured with an HQ 40d multi-parameter, along with PHC3OH, LDO101, and MTC101 probes supplied by the HACH company, Loveland, CO, USA. Ammonium nitrogen (NH_4-N), nitrite nitrogen (NO_2-N), nitrate nitrogen (NO_3-N), total nitrogen (TN), total phosphorus (TP), and chemical oxygen demand (COD) concentrations of the influent and effluent samples were measured with an ultraviolet–visible (UV-VIS) HACH DR 6000 spectrophotometer, HACH DRB 200 reactor blocks (supplied by HACH company, USA), and a Kjeldahl digestion–distillation unit (supplied by VELP Scientifica, Usmate Velate, Italy), following the protocols described by the instrument manuals. The five-day biochemical oxygen demand (BOD_5) concentration of the wastewater samples was quantified with HACH BOD TRAK II manometric instruments and incubators (supplied by HACH company, USA) operated at 20 °C following the protocols described by the instrument's manual. The coliform numbers of the wastewater samples were quantified with Macconkey agar and an incubator with an operational temperature of 37 °C.

Canna indica plants were harvested from the two systems after the termination of the experiment. The harvested plants were divided into aboveground (AG) and underground (UG) parts and oven-dried at a temperature of 80 °C until a constant dry biomass weight was achieved. The used stone dust media were collected from the first compartment (normal or electrode-integrated upflow-based wetland) in system 1 or 2; the used stone dust media were also collected from the three segments of the second compartment (surface flow wetland) in system 1 or 2. The nitrogen and phosphorus concentration of the plant biomass, unused (fresh) and used stone dust media were measured according to the protocols of digestion–distillation and vanadomolybdo phosphoric yellow color methods [22].

3. Results and Discussion

3.1. Organic Matter Removal

Figure 2 represents mean effluent organic (BOD and COD) concentration and removal efficiency (in percentages) as the leachate wastewater passed through the first (normal/electrode-integrated upflow-based wetland) and second compartment (surface flow wetland) in system 1 or 2. The normal upflow-based wetland (i.e., first compartment) in system 1 produced mean effluent BOD and COD concentration ranges of 72–120 mg/L and 122–262 mg/L, respectively, within Phases I and II. Mean effluent BOD and COD concentration ranges with the electrode-integrated upflow-based wetland (i.e., first compartment) in system 2 were 69–94 mg/L and 134–256 mg/L, respectively, within Phases I and II.

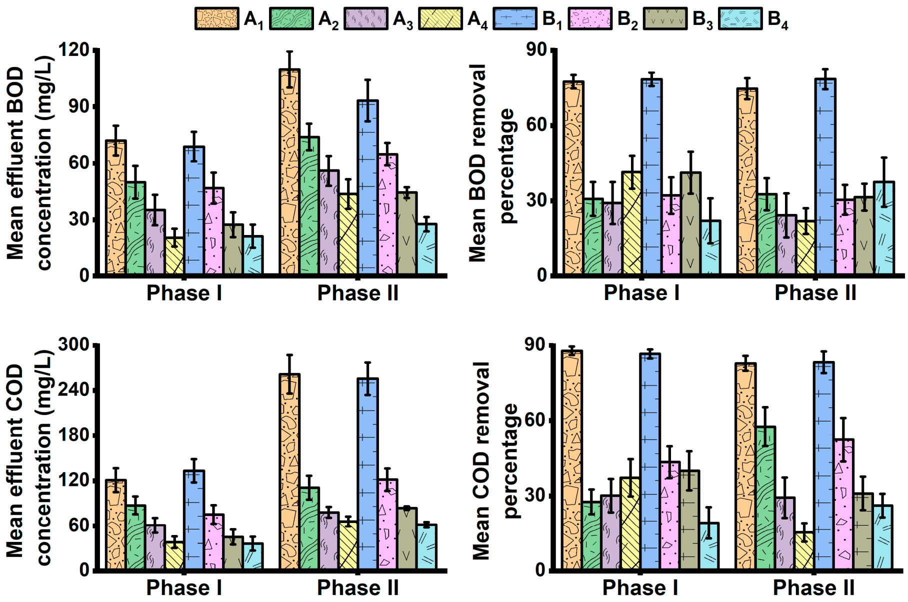

Figure 2. Mean effluent organic (BOD and COD) concentration produced across the two systems and removal efficiency within Phases I and II. Bars indicate standard error. System 1: sample collection point A_1 (outlet of the normal upflow-based wetland), sample collection points A_2, A_3, and A_4 (first, second, and third segments, respectively, of the surface flow wetland). System 2: sample collection point B_1 (outlet of the electrode-integrated upflow-based wetland), sample collection points B_2, B_3, and B_4 (first, second and third segments, respectively, of the surface flow wetland).

Organic removal in normal wetlands is primarily achieved by electrochemically inactive aerobic and anaerobic routes [40,41]. On the other hand, electrochemically active and inactive removal pathways coexist in electrode-integrated wetlands that improve their overall organic removal performances [32,42,43]. Lower mean effluent organic concentration ranges of the electrode-integrated upflow-based wetland compared to the normal upflow-based wetland also support the positive influence of electrode integration and associated bioreactions on overall organic removal performances.

The mean effluent organic concentration across the normal upflow-based wetland (in system 1) increased during Phase II (compared to Phase I performances); such differences were statistically significant ($p < 0.05$). Effluent redox profiles decreased sharply, i.e., from 83 mV (Phase I) to 27 mV (Phase II) across the normal upflow-based wetland (Supplementary Material S1). Such a sharp decrease in effluent redox potential (in Phase II) denotes the development of a more anoxic gradient inside the media pores of the normal upflow-based wetland. Input COD loading across both systems (of this study) increased, i.e., from 40 g/m^2d (Phase I) to 285 g/m^2d (Phase II), primarily because of hydraulic load increment during the latter operational period. Such input load increment in Phase II could have intensified influent organic removal via aerobic routes and associated rapid available oxygen depletion [35,44] inside the media of the normal upflow-based wetland. These interlinked phenomena might have suppressed aerobic organic removals, thus resulting in a statistically significant ($p < 0.05$) effluent organic concentration increase in Phase II (compared to Phase I performance), along with redox potentials decrease with the normal upflow-based wetland. The mean effluent redox concentration across the electrode-integrated upflow-based wetland (in system 2) also decreased because of input load increment, i.e., from 79 mV (Phase I) to 75 mV (Phase II). However, such a transition was not as sharp as observed with the normal upflow-based wetland. Electrochemical organic matter oxidation could have minimized electrochemically inactive aerobic route-based rapid oxygen consumption, particularly in Phase II (when the overall input load increased) in the case of electrode-integrated upflow-based wetlands. This contribution was probably the primary factor that resulted in insignificant effluent organic concentration differences ($p > 0.05$) produced by the electrode-integrated upflow-based wetland between the two phases.

Mean effluent organic concentration profile ranges in the three segments of the surface flow wetland in system 1 were 20–74 mg/L BOD and 38–111 mg/L COD within Phases I and II. The mean effluent organic concentration profile ranges in the three segments of the surface flow wetland in system 2 were 18–65 mg/L BOD and 36–122 mg/L COD within Phases I and II. Organic removal in water column-based wetlands is achieved through particulate sedimentation, filtration, and microbial decomposition induced by the water column, media, and plants [41,45,46]. Overall mean effluent redox profiles in the three segments of surface flow wetlands (in both systems) ranged between 50 and 138 mV (within Phases I and II: Supplementary Material S1), which exceeded the redox profiles of the preceding upflow-based wetlands. Effluent redox potential increment across the surface flow wetlands could be primarily attributed to organic matter removal in the preceding upflow-based wetlands that prevented more anoxic/anaerobic gradient development in the water column of surface flow wetlands. Such environmental conditions could have favored microbial-based decomposition via aerobic routes. Moreover, flow interception by the baffle walls and associated forced wastewater passing through the media could have reinforced media-based physicochemical and microbial organic removal pathways [30]. This is also reflected by a gradual organic concentration decrease (Figure 2) within the subsequent segments of the surface flow wetlands.

The surface flow wetland in system 1 produced higher mean effluent organic concentration ranges compared to the surface flow wetland in system 2. Statistically significant ($p < 0.05$) effluent organic concentration increment due to input load increment in Phase II (compared to Phase I performance) was observed with the surface flow wetland in system 1. On the other hand, mean effluent organic concentration differences (between the two phases) of the surface flow wetland in system 2 were not statistically significant ($p > 0.05$). These trends coincide with the performance of the preceding normal and electrode-integrated upflow-based wetlands. Hence, it could be stated that the organic matter composition of the effluent produced by the upflow-based wetlands heavily influenced the organic removal performance of the subsequent surface flow wetlands.

It should be noted that the surface flow wetlands in both systems of this study achieved mean BOD and COD removal efficiency ranges of 65–70% and 72–75%, respectively, within the two operational periods. These data profiles were beneath the mean BOD and COD removal efficiency ranges, i.e., 76–79% and 84–86%, respectively, of the preceding upflow-based wetlands. A similar trend was reported previously: a mean BOD removal of 69% and 19% in the first stage vertical flow and the following surface flow wetland, respectively, by Nguyen, Nguyen [45], or a mean COD removal of 65% and 25% in the first stage vertical flow and the second stage surface flow wetland, respectively, by Nguyen, Tran [47]. The authors attributed such performance deviation between the two stages to the absence of a filter layer in the surface flow wetlands that diminished media-based physicochemical and microbial-based organic removal. In this study, although the mean organic removal efficiency of the surface flow wetlands (in both systems) was beneath the preceding upflow-based wetlands, these profiles exceeded the performances of the previously reported surface flow wetlands. The presence of baffle walls and associated upward–downward flow directions were probably the primary factor that strengthened the water column and plant- and media-based physicochemical and microbial removal pathways.

3.2. Nutrient Removal: Partitioning between Wetland Plants and Media

3.2.1. Mean Effluent Concentration

Figure 3 represents mean effluent nutrient concentration and removal efficiency as the leachate wastewater passed through the first (normal/electrode-integrated upflow-based wetland) and second compartment (surface flow wetland) in system 1 or 2. The normal upflow-based wetland (in system 1) produced mean effluent NH_4-N, NO_3-N, TN, and TP concentration ranges of 17–20 mg/L, 47–79 mg/L, 64–118 mg/L, and 39–60 mg/L, respectively, during Phases I and II. The electrode-integrated upflow-based wetland (in system 2) produced mean effluent concentration ranges of 14–18 mg/L, 39–60 mg/L, 60–96 mg/L, and 44–70 mg/L, respectively, in Phases I and II.

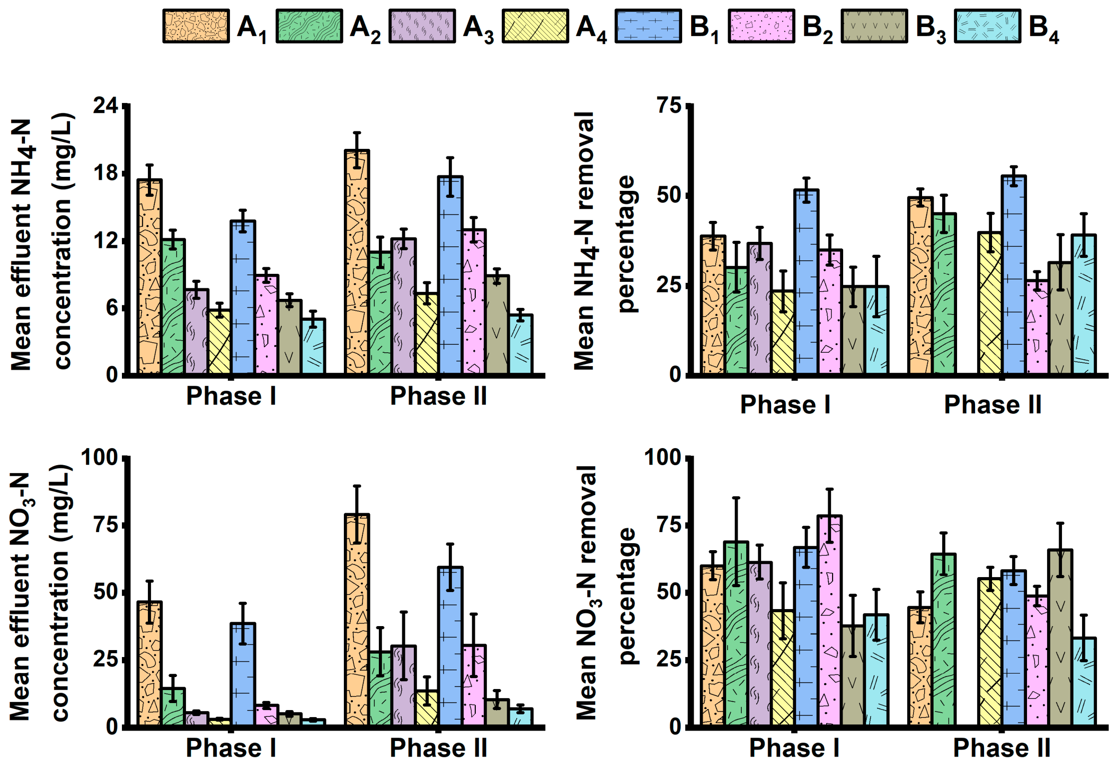

Figure 3. *Cont.*

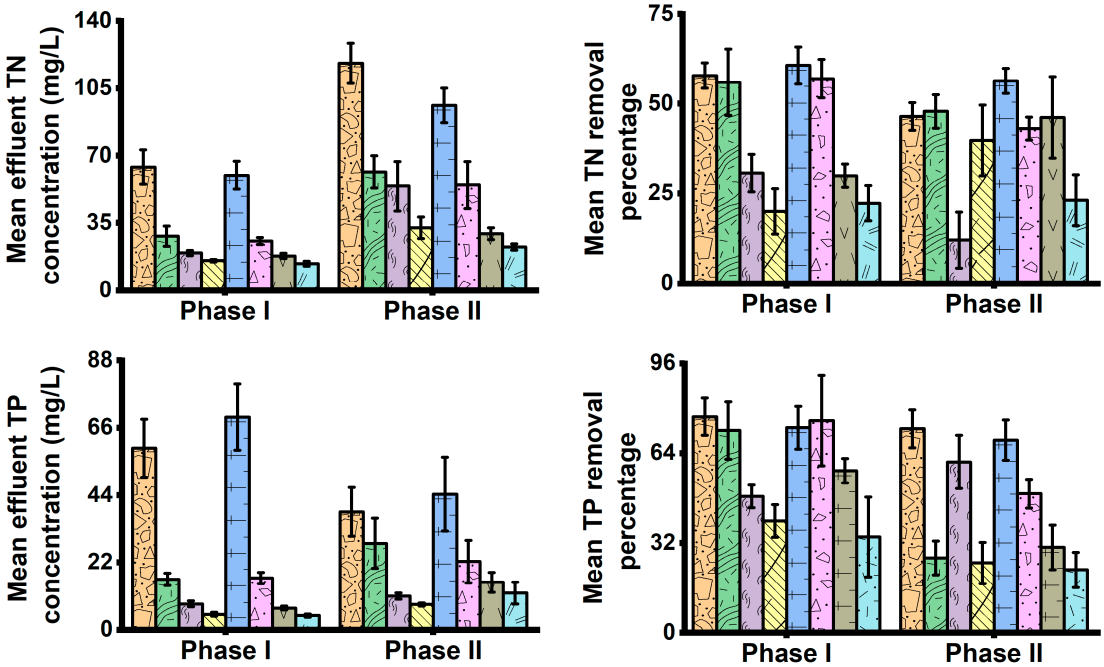

Figure 3. Mean effluent nutrient concentration produced across the two systems and removal efficiency within Phases I and II. Bars indicate standard error. System 1: sample collection point A_1 (outlet of the normal upflow-based wetland), sample collection points A_2, A_3, and A_4 (first, second, and third segments, respectively, of the surface flow wetland). System 2: sample collection point B_1 (outlet of the electrode-integrated upflow-based wetland), sample collection points B_2, B_3, and B_4 (first, second and third segments, respectively, of the surface flow wetland).

The mean effluent NH_4-N, NO_3-N, TN, and TP concentration ranges within the three segments of the surface flow wetland in system 1 were 5–13 mg/L, 3–31 mg/L, 15–62 mg/L, and 5–29 mg/L, respectively, in Phases I and II. Regarding system 2, mean effluent concentration ranges within the three segments of the surface flow were 4–12 mg/L, 3–32 mg/L, 14–55 mg/L, and 4–23 mg/L, respectively, in Phases I and II.

Wastewater nutrient, i.e., nitrogen and phosphorus, removal with constructed wetlands is achieved through three pathways: plant uptake, media-based adsorption, and microbial decomposition [48–51]. The probable contribution of these distinct pathways on observed nutrient removals (with the two systems), and the factors that could have assisted such removals are discussed in the following paragraphs.

3.2.2. Plant Uptake

Wastewater nutrients often accumulate in plant tissues through uptake pathways in wetland systems [48,52,53]. Hence, nutrient concentration profiles of the aboveground (AG) and underground (UG) biomass of Canna indica (planted into the two compartments of both systems), along with plant-based nutrient accumulation percentages in each of the two compartments (of a single system) with respect to corresponding overall removal, were quantified and summarized in Table 2. Nitrogen accumulation percentage in AG or UG biomass of normal and electrode-integrated upflow-based wetlands was lower; accumulation percentages with the plant tissues of the electrode-integrated upflow-based wetland were slightly higher than the normal upflow-based wetland. Higher influent nitrogen concentration across the upflow-based wetlands could have exceeded the nitrogen uptake capacities of the plants [50], thus resulting in comparatively lower accumulation percentages in plant tissues of the upflow-based wetlands.

Table 2. Nitrogen (N) and phosphorus (P) accumulation concentration (mg/kg) and percentages (%) in aboveground (AG) and underground (UG) plant tissues in systems 1 and 2.

Systems	Compartments	Components	N Accumulation (mg/kg)		P Accumulation (mg/kg)		N Accumulation (%)		P Accumulation (%)	
			AG	UG	AG	UG	AG	UG	AG	UG
1	First (upflow)	Normal	13,000	18,000	1300	3400	0.6	0.5	0.0001	0.0002
	Second (surface flow)	Segment 1	21,000	20,000	600	1700	5.5	3.1	0.0005	0.0008
		Segment 2	24,000	25,000	800	1300	4.9	2.9	0.001	0.001
		Segment 3	16,000	23,000	1000	1700	6.5	4.4	0.005	0.004
2	First (upflow)	Electrode	15,000	23,000	1200	1600	2.2	1.5	0.0003	0.0002
	Second (surface flow)	Segment 1	15,000	19,000	800	1000	5.8	3.4	0.0008	0.0004
		Segment 2	17,000	24,000	1100	1300	12.1	9.9	0.005	0.003
		Segment 3	18,000	23,000	700	1300	25.1	17.0	0.009	0.009

The nitrogen accumulation percentage of the AG or UG plant biomass harvested from the three segments of surface flow wetlands was higher compared to those of the upflow-based wetlands. Nitrogen accumulation percentages were maximum in the plant tissues of the last segments (of both surface flow wetlands) compared to the profiles of the preceding segments. Nitrogen removal in the preceding upflow-based wetlands and additional gradual nitrogen concentration decreases in wastewater within the subsequent segments (Figure 3) were probably major factors that increased nitrogen accumulation percentages in the plant tissues of the surface flow wetlands. These findings agree with Vymazal [48], who reported more efficient performance of wetland plants to remove nutrients under low loading conditions. In general, low and partial nitrogen accumulation profiles in plant tissues (with respect to observed removal) of the upflow-based and surface wetlands, respectively, suggest the probable influence of other removal pathways, such as media-based adsorption on observed nitrogen removal (Figure 3), and will be discussed in the following media-based adsorption subsection.

The phosphorus accumulation percentage with the AG or UG plant biomass of the upflow-based and surface flow wetlands (of the two systems) was very low, probably due to substantial phosphorus presence in the influent leachate wastewater (Table 1). Therefore, plant uptake was not the major kinetic that contributed to overall observed phosphorus removals (Figure 3). Chemical pathways induced by the media often favor phosphorus removal in wetland systems [54–56]. The probable influence of such chemical pathways on observed phosphorus removal in the two systems of this study is discussed in the following media-based adsorption subsection.

3.2.3. Media-Based Adsorption

The chemical composition of the wetland media often provides favorable conditions for removing incoming nutrients through adsorption [22,49]. To assess the probable existence of such chemical-based pathways, nutrient concentration profiles of the unused (fresh) and used (collected from the upflow-based wetlands and the three segments of the surface flow wetlands) stone dust media were measured. Table 3 provides the nutrient concentration data profiles of the unused and used media. As observed in Table 3, the nitrogen concentration of the used stone dust media of upflow-based and surface flow wetlands in both systems exceeded the profiles of the unused media. Such nitrogen concentration increment with the used media indicates the probable existence of media-based chemical adsorption pathways in both systems of this study.

Wetland media-oriented nitrogen removal is triggered by specific cations such as potassium (K^+), sodium (Na^+), calcium (Ca^{2+}), and magnesium (Mg^{2+}). The presence of these cations in the wetland media assists in forming cation exchange sites on the media surface. Wastewater NH_4-N could be adsorbed within these cation exchange sites, thus contributing to incoming nitrogen removal [57,58]. Energy-Dispersive Spectroscopy (EDS) analysis of the unused and used (collected from the upflow-based wetlands and the three segments of the surface flow wetlands) media was also conducted in this study (Supplementary Material S2) to investigate the probable presence of the specific cations that might have supported chemical-based adsorption with the two systems in this study. As observed in the data profiles produced from EDS analysis, the unused and used stone dust media (employed as wetland media in this study) were probably composed of K, Ca, Na, and Mg. The presence of such cations might have promoted NH_4-N adsorption, which is further supported by media concentration profile analysis, i.e., nitrogen concentration increment of the used media compared to unused media profiles (Table 3).

Table 3. Nitrogen (N) and phosphorus (P) composition profiles of the unused and used stone dust media.

Systems	Compartments	Components	N Concentration of Media (mg/kg) Unused	N Used	P Concentration of Media (mg/kg) Unused	P Used
1	First (upflow)	Normal	1000	1200	1100	1600
	Second (surface flow)	Segment 1		1100		100
		Segment 2		2100		100
		Segment 3		1500		600
2	First (upflow)	Electrode	1000	1400	1100	1200
	Second (surface flow)	Segment 1		1200		900
		Segment 2		1300		1500
		Segment 3		1200		700

The phosphorus concentration of the used media of normal and upflow-based wetlands exceeded the unused media profiles (Table 3), demonstrating the probable contribution of chemical adsorption on observed phosphorus removal (Figure 3) with upflow-based wetlands. Chemical-based phosphorus adsorption, primarily induced by calcium (Ca), aluminum (Al), and iron (Fe) composition of the media, is often reported as a major removal route with constructed wetlands [49,55]. EDS analysis (Supplementary Material S2) suggests the probable presence of these elements with the unused and used stone dust media that could have supported phosphorus adsorption in upflow-based wetlands. The used stone dust media extracted from the three segments of both surface flow wetlands demonstrate an opposite trend; phosphorus concentration in these used media was beneath the unused media profile. Effluent phosphorus concentration decreases compared to the influent profiles (Figure 3 and Table 1), but higher phosphorus concentration of the unused media (than the used media of the surface flow wetlands-Table 3) undermines the positive impact of chemical pathways on observed phosphorus removals. Such contradictory trends between influent–effluent and media-based phosphorus concentration profiles of the surface flow wetlands suggest the probable existence of other removal pathways. Although such a discrepancy between water quality and media-based concentration profiles was not observed in the case of the other nutrient, i.e., nitrogen, microbial nitrogen decomposition has been reported as a major nitrogen removal pathway that could coexist with chemical-based removal in the case of constructed wetlands [36,50]. Hence, additional nutrient removal analysis within the two systems has been presented in Section 3.3 to elucidate the probable contribution of other pathways and factors on observed nutrient removals.

3.2.4. Physical Properties of the Media

The physical structures of the unused and used stone dust materials (employed as the main wetland media in this study) have been captured by scanning electron microscopy (SEM) images and presented in Figure 4. According to these images, the surface area of the unused (fresh) stone media was relatively smooth. On the other hand, the surface area of the used media (extracted from the upflow-based wetlands and the segments of the surface flow wetlands) was covered with a thick layer, along with aggregate formation [59]. Biofilm development, organic particulates, and root fragment deposition probably contributed to composite layer formation on the surface area of the used media [60]. Microbial decomposition of organics (discussed in Section 3.1) and nutrients (will be discussed in Section 3.3) could have existed in the aerobic and anaerobic pores of the formed biolayer, thus contributing to overall pollutant removal [56] along with plant uptake and media-based adsorption.

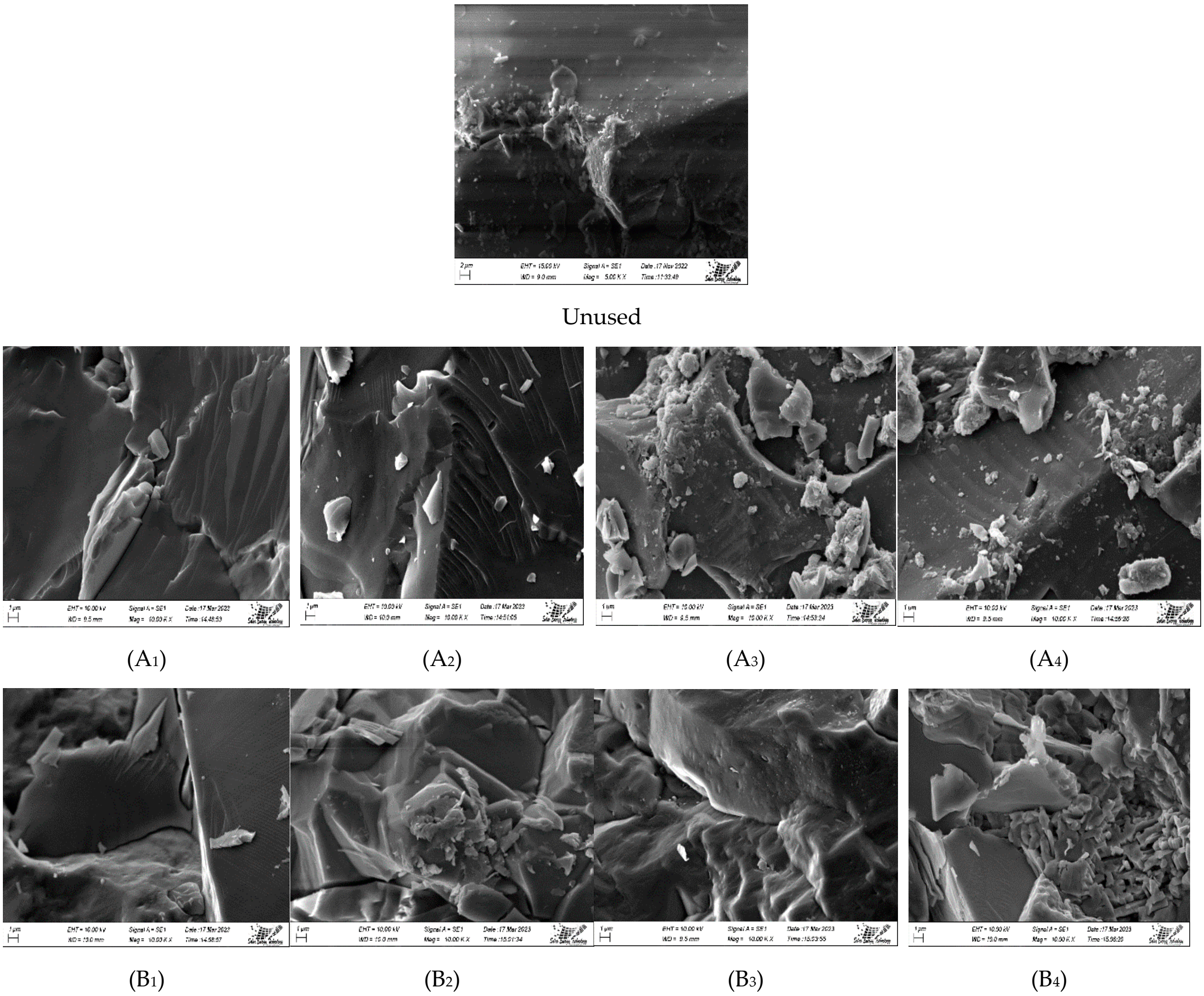

Figure 4. SEM images of the unused and used stone dust media. System 1: (**A_1**) used media collected from the normal upflow-based wetland; (**A_2–A_4**) used media collected from the first, second, and third segments, respectively, of the surface flow wetland. System 2: (**B_1**) used media collected from the electrode-integrated upflow-based wetland; (**B_2–B_4**) used media collected from the first, second, and third segments, respectively, of the surface flow wetland.

3.3. Synthesis of Nutrient Removal

Plant-based nitrogen accumulation percentage data profiles (Table 2) specify their minor (in case of upflow-based wetlands) or partial (in case of surface flow wetlands) contributions to overall nitrogen removal. It was unlikely that media-based NH_4-N adsorption (Table 3) primarily contributed to observed TN removals in both systems (Figure 3), as NO_3-N was the major nitrogenous component in the leachate wastewater (Table 1). In constructed wetlands, NO_3-N is primarily removed through denitrification (NO_3-N reduction to N_2 gas) and often coexists with microbial nitrification (conversion of NH_4-N to NO_2-N and then to NO_3-N) [26,40,61]. The decrease in pH across the effluent of upflow-based and surface flow wetlands (Supplementary Material S1) suggests the contribution of nitrification (along with media-based NH_4-N adsorption) because of its linkage with wastewater alkalinity consumption [62]. Effluent redox potential increment across the upflow-based and surface flow wetlands (Supplementary Material S1) compared to influent profiles (Table 1) signifies the presence of favorable environmental conditions for the progression of nitrification (inside the media pores) due to its dependency on dissolved oxygen availability [63].

The mean NO_2-N concentration in the leachate wastewater was very low (Table 1) and was further reduced in the effluent in the two systems (i.e., ranging between 0.05 and 0.1 mg/L). The substantial NO_3-N concentration of the leachate wastewater (Table 1) dropped sharply in the effluent produced by the upflow-based and surface wetlands (Figure 3). Effluent NO_2-N and NO_3-N concentration reduction (along with TN concentration decrease) across the upflow-based and surface flow wetlands depicts a major contribution from microbial denitrification in removing the overall nitrogen composition of the landfill leachate. Such microbial pathways heavily rely on the availability of organic carbon that is provided by wastewater and employed media in constructed wetlands [12,61,64]. The organic biodegradation of the leachate wastewater was very low (Table 1); therefore, wastewater-based carbon contribution to support denitrification could be ruled out. EDS analysis suggests the probable presence of organic carbon (as a chemical ingredient) in the unused and used stone media (Supplementary Material S2). The microbial community of the formed biolayer on media surfaces (Figure 4) might have utilized such available media-based carbon to reduce NO_x components in deep anoxic/anaerobic portions.

The electrode-integrated upflow-based wetland (in system 2) produced lower effluent TN concentration ranges compared to normal upflow-based wetlands (in System 1) within both operational phases. Previous studies reported a positive impact of electrode integration on overall nitrogen removal performances due to electrode-based NH_4-N oxidation and additional electron production from electrochemical organic matter oxidation to support denitrification [33,34]. Effluent TN concentration ranges across the surface flow wetland (in system 2) were lower than the surface flow wetland (in system 1) during the two operational periods. Nitrogen accumulation percentage in plant tissues, particularly with the last two segments in the surface flow wetland (in system 2), was higher than those in the surface flow wetland (in system 1) due to receiving lower TN concentrations from the preceding electrode-integrated upflow-based wetland and gradual nitrogen concentration decrease in the three segments. The synergetic impact of these factors could have improved the overall TN removal of the surface flow wetland in system 2. Effluent TN concentration increased across the upflow-based or surface flow wetlands in both systems in Phase II compared to the corresponding Phase I performance, which was statistically significant ($p < 0.05$). Such differences indicate the adverse impact of load increment on the nitrogen removal routes: plant uptake, media-based adsorption, and microbial decomposition.

Regarding phosphorus removal performance, plant-based accumulation percentages (with the upflow-based and surface flow wetlands—Table 2) were negligible. The phosphorus concentration increment of the used media in the upflow-based wetlands exceeded those of the unused media (Table 3). This a trend indicates the influence of media-based phosphorus adsorption in reducing effluent phosphorus concentration. In contrast, there is a lower phosphorus concentration in the used media (compared to unused media profiles) in the three segments in surface flow wetlands, but the effluent phosphorus concentration decreased (Figure 3), suggesting the probable contribution of other removal routes that could have resulted in phosphorus disappearance in both systems. A recent study by Wu, Wang [65] reported the contribution of microbial decomposition in removing influent phosphorus with constructed wetlands; molecular analysis was presented in that study to support the findings. Such a route might have influenced the disappearance of phosphorus from the surface flow wetlands of this study. However, this hypothesis could not be confirmed due to a lack of evidence from molecular analysis. TP concentration differences across the upflow-based or surface flow wetlands between Phase I and II in both systems were not statistically significant ($p > 0.05$), specifying stable removal despite input load variations.

The mean TN and TP removal efficiency of the upflow-based wetlands were 50–59% and 71–76%, respectively. The surface flow wetlands achieved 75–77% and 86–88% removal, respectively. Higher nutrient removal efficiency in the surface flow wetlands compared to the preceding upflow-based wetlands further signifies the influence of controlled flow direction on the nutrient removal pathways. The nitrogen removal efficiency in the surface flow wetlands in this study exceeded the mean 58% NO_3-N removals in the second stage surface flow wetland of a hybrid wetland reported by Nguyen, Tran [47]. However, the reported nutrient removal ranges were below those, i.e., 83–91% (TN) and 92–100% (TP), in the second stage surface flow wetland in a hybrid system reported by Saeed, Miah [21].

3.4. Coliform Removal

Figure 5 represents the mean effluent coliform number and removal efficiency as the leachate wastewater passed through the first (normal/electrode-integrated upflow-based wetland) and second compartment (surface flow wetland) in system 1 or 2. The mean effluent coliform number ranges with the normal and electrode-integrated upflow-based wetlands were 15,222–16,500 CFU/100 mL and 12,889–13,333 CFU/100 mL, respectively, during the two operational phases. Coliform mortality with constructed wetlands is primarily achieved by three distinct routes: physical (sedimentation, filtration, and UV radiation), chemical (toxin production from plants, adsorption, and oxidation), and biological (microbial predation and natural die-off) [66,67]. Moreover, the current generation between the electrodes could improve coliform mortality with bioenergy-producing wetlands [43]. This supplementary advantage might have produced lower effluent coliform number ranges across the electrode-integrated upflow-based wetland compared to the normal upflow-based system within the two operational periods.

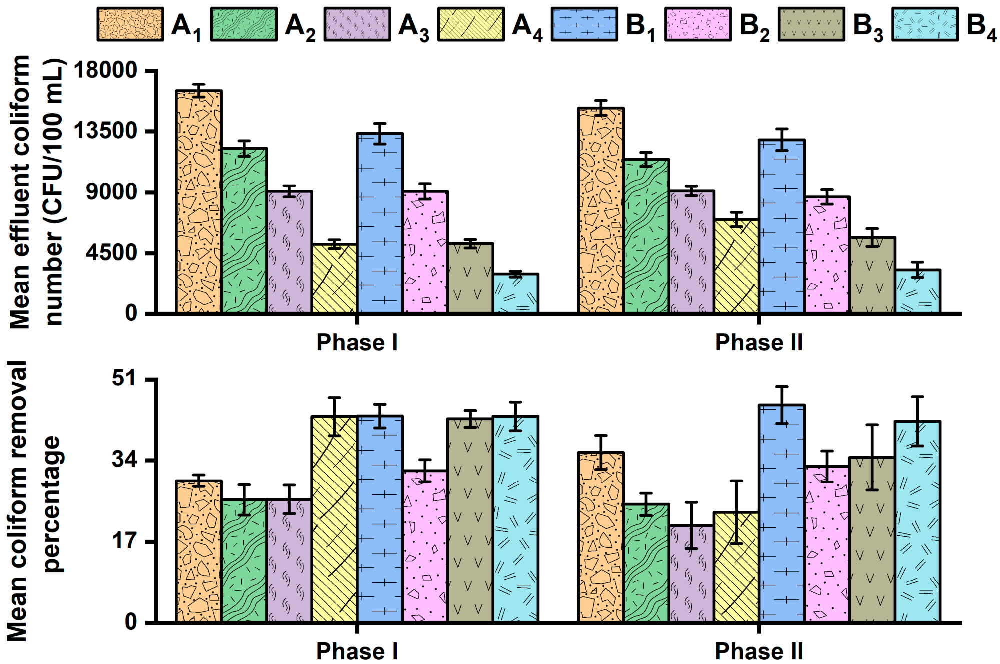

Figure 5. Mean effluent coliform number produced across the two systems and removal efficiency within Phases I and II. Bars indicate standard error. System 1: sample collection point A_1 (outlet of the normal upflow-based wetland), sample collection points A_2, A_3, and A_4 (first, second, and third segments, respectively, of the surface flow wetland). System 2: sample collection point B_1 (outlet of the electrode-integrated upflow-based wetland), sample collection points B_2, B_3, and B_4 (first, second and third segments, respectively, of the surface flow wetland).

The mean effluent coliform number ranges across the surface flow wetlands in systems 1 and 2 were 5166–12,250 CFU/100 mL and 2958–9084 CFU/100 mL, respectively, within the two operational periods. The mean effluent coliform number in all segments of the surface flow wetland in system 2 was beneath those of the surface flow wetland in system 1. Lower effluent coliform numbers of the electrode-integrated upflow-based wetland could have intensified coliform removal routes in the subsequent surface flow wetland (in system 2). Effluent coliform concentration differences across the upflow-based or surface wetlands were not statistically significant ($p > 0.05$) between Phases I and II, signifying stable removal performance despite unstable loading conditions.

Regarding removal efficiency, 32–63% and 44–77% coliform removals were observed within both compartments in systems 1 and 2, respectively. The surface flow wetlands achieved higher removal efficiency (than the upflow-based wetlands) primarily because of two synergistic impacts: (a) coliform mortality in the preceding upflow-based wetlands; and (b) upward–downward flow direction induced by the baffle walls that triggered physical, chemical, and biological coliform removal routes in surface flow wetlands. Overall, the mean coliform removal efficiency with systems 1 and 2 was 75 and 87%; these data profiles signify better coliform removal performance in system 2. Therefore, electrode integration in the upflow-based wetland positively influenced coliform mortality of the whole system.

3.5. Energy Production

Figure 6 illustrates bioenergy, i.e., voltage and power density production of the electrode-integrated upflow-based wetland (in system 2) with respect to time (per day expressed as minutes). The voltage production ranged between 96 and 139 mV in Phase I and 118 and 193 mV in Phase II. Power density production ranged between 235 and 487 mW/m^3 in Phase I and 354 and 946 mW/m^3 in Phase II. These data ranges depict higher bioenergy production of the electrode-integrated upflow-based wetland in Phase II compared to Phase I performances. Such bioenergy production improvement (during Phase II) might be linked to input COD load increment and associated intensified electrochemical organic matter oxidation, electron production, and power generation; the performance of the electrode-integrated wetlands heavily relies on organic load rates [32].

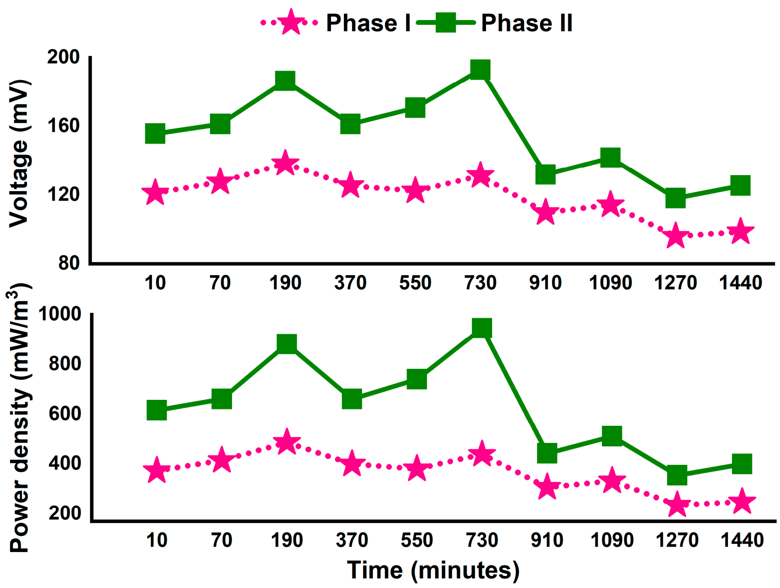

Figure 6. Voltage and power density production of the electrode-integrated upflow-based wetland (in system 2) during the two operational phases.

Internal resistance development between the electrodes often interrupts the operational performance of the electrode-integrated constructed wetlands [44]. Such performance interrupting factor is assessed by a polarization test that involves the integration of external resistors (of different resistance values) with the bioenergy-producing wetlands and quantifying variable voltage, power density, and current density production [22]. This study conducted a polarization test to assess the probable internal resistance development with the electrode-integrated upflow-based wetland (in system 2) by connecting resistors with different resistance values (ranging between 100 and 33,000 Ω). The variable voltage, current, and power density produced by the polarization test are presented in Figure 7, often referred to as a polarization curve. According to Figure 7, a maximum power density of 167 mW/m^3 was produced during the polarization test when the electrode-integrated upflow-based wetland was connected to an external resistor of 330 Ω. As such, internal resistance developed with the electrode-integrated system in this study.

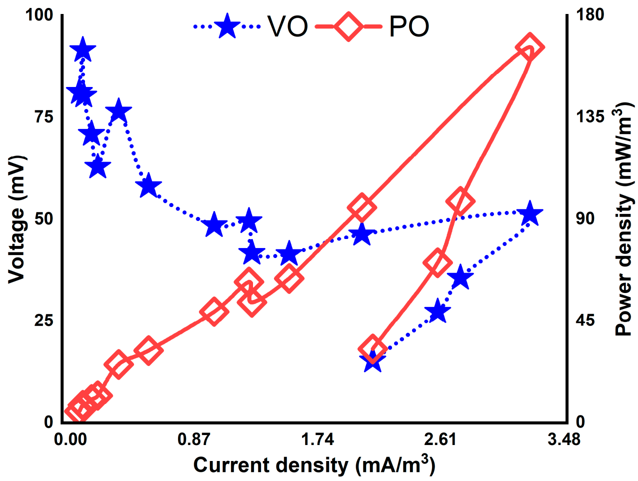

Figure 7. Voltage, current, and power density plots produced from the polarization test. The symbols VO and PO represent voltage and power density, respectively.

3.6. Research Implication

Table 4 summarizes the organic and nutrient performances of the integrated wetlands employed previously for the treatment of different real wastewater. The overall mean organic and nutrient removal efficiency of the leachate treatment-based integrated upflow–surface flow wetlands without (system 1) and with (system 2) electrode coupling in this study are also summarized in Table 4. As observed in Table 4, the pollutant removal performances of the developed integrated wetlands in this study exceeded the performances of the previously reported integrated wetlands. Moreover, the overall mean organic and nitrogen removal efficiency in the two integrated systems (in this study) were above the reported mean ranges achieved by the leachate treatment-based normal hybrid wetlands employed worldwide [8]. Therefore, the integrated wetland systems (in this study) could be an attractive option for raw landfill leachate or other wastewater treatment, particularly in compacted areas where land acquisition for wetland construction could substantially increase operational costs.

Table 4. Organic and nutrient removal performances of the integrated constructed wetlands employed previously and in this study.

Source	Influent	Integrated Wetland Type	Removal Efficiency (%)		
			Organic	Nitrogen	Phosphorus
Liu, Sun [37]	Swine wastewater	Electrode-coupled downflow–upflow wetlands	82–88	64–77	-
Meng, Feng [29]	Contaminated lake	Vertical baffled flow wetlands	83	28	59
Zhao, Zhao [28]	Highway runoff	Horizontal flow wetlands with internal baffle walls	68	78	64
Cui, Ouyang [23]	Septic tank effluent	Vertical flow, horizontal flow, and hybrid flow-based wetlands with baffle walls	90	62	95
Saeed, Al-Muyeed [31]	Municipal	Vertical followed by horizontal flow wetlands with internal baffle walls	77–83	12–56	37–64
Tee, Lim [24]	Domestic	Horizontal flow wetlands with baffle walls	59–79	>98	-
This study	**Landfill leachate**	**Integrated upflow–surface flow wetlands with or without electrode coupling**	**91–97**	**88–91**	**96–97**

Lower effluent organic and nitrogen concentration production in system 2 (including electrode-integrated upflow-based wetlands) than the other system integrated with normal upflow-based wetlands indicates the positive impact of electrode coupling for producing higher effluent quality. Future works should focus on assessing the pollutant removal performances of the developed leachate wastewater treatment-based integrated constructed wetlands (without and with electrode coupling) under wider loading deviation ranges that might demonstrate their resilience capacities. In addition, the impact of other types of media in supporting physicochemical removal routes and microbial decomposition should also be investigated by employing developed leachate treatment-based wetlands to produce higher effluent quality.

4. Conclusions

The main conclusions of this study are summarized below.

The developed integrated wetlands in this study (without and with electrode coupling) achieved a mean of 91–97% organic, 88–91% nitrogen, and 96–97% phosphorus removals.

Both systems achieved comparatively stable nutrient removals compared to organic removal profiles under the examined loading ranges. Controlled flow direction induced by the baffle walls of the surface flow wetlands improved overall pollutant removals in both systems.

Organic removal performance depended on input loading rate variations in the case of the system integrated with normal upflow-based and surface flow wetlands. Such a dependency between organic removal and input load variations was counterbalanced with the electrode-dependent system due to the synergistic effect of electrochemically active and inactive organic removal pathways.

Nitrogen removal was heavily dependent on denitrification with both systems; media-based adsorption contributed to nutrient removal. Electrochemical-based bioreactions improved nitrogen removal in the electrode-coupled system compared to the normal system, i.e., without electrode integration.

Nitrogen accumulation in the plant tissues of surface flow wetlands ranged between 3 and 25%, reflecting the dominance of the biological pathway on nitrogen removal (in surface flow wetlands). Such a pathway did not influence overall phosphorus removal.

Power density production of the electrode-coupled system ranged between 235 and 946 mW/m^3. Bioenergy production and input organic load increment were positively correlated.

This study provides the potential application of the integrated upflow-based and surface flow wetlands and the positive impact of electrode integration (in such a system) to achieve better pollutant removal from landfill leachate under the applied loading ranges.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/w16131776/s1, Supplementary material S1: Figure. Mean effluent pH and redox (Eh) concentration profiles produced across the two systems during Phases I and II. Supplementary material S2: Table. Probable elemental composition percentages of the unused and used stone dust media as indicated by the EDS analysis.

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Conflicts of Interest: The authors declare no conflict of interest.

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