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Hybrid constructed wetland for treatment of power plant effluent polluted with hydrocarbons

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ABSTRACT

Constructed wetlands (CWs) have good potential for application in the treatment of various types of polluted water in Iraq, including industrial wastewater, due to their low cost relative to the current conventional treatment technologies. Hybrid CW with a continuous-flow system was tested for the treatment of highly polluted industrial wastewater from the Al-Najibiyia Power Plant in Basrah City for 72 days with 5 days of retention time. The system consisted of a horizontal subsurface flow system (HSSF) planted with *Phragmites australis* (Cav.) followed by a surface flow (SF) system planted with *Hydrilla verticillata* (L.f.) Royle. A second system without plants served as a contaminant control system. Total petroleum hydrocarbons (TPHs), petroleum aromatic hydrocarbons (PAHs) and water quality were measured for both raw and treated wastewaters and plant status parameters were monitored. The presence of both plants and substrate had a significant effect on the removal efficiency and mass removal rate of all pollutants at ($p < 0.05$). The planted system performance showed a percentage removal of pollutants reaching to: 65.9 % for BOD_5, 91.0 % for COD and 88.8 % for total suspended solids (TSS) while removal efficiency of BOD_5, COD and TSS for unplanted systems was only 44.7, 64.5 and 68.7 %, respectively. The removal efficiencies of TPHs, and PAHs by the hybrid system were 96.5, and 96.0 %, compared to only 71.0 % and 51.0 % for unplanted system, respectively, showing that hybrid CW could enhance pollutant removal efficiency better than single CW. It is revealed also that the high efficiency in removing pollutants is the result of the plant and substrate presence and the interaction between plants and the surrounding microbial community. Thus, constructed wetlands (CWs) are a good option for industrial wastewater treatment due to its simple construction and effectiveness to remove hydrocarbons.

1. Introduction

All over the world, petroleum and its refined products are the main source of energy in industry and in daily life. Hydrocarbons enter the environment through many routes, such as oil spillage, electronic and medical waste, industrial processes, landfills associated with petroleum production, pesticides, combustion of fossil fuel, or as by-products from commercial or private uses [1–3]. Furthermore, hydrocarbons contamination may occur in effluent from power plants. As a nearest example, our present study used effluent from Al-Najibiyia in Basrah city in Iraq containing a wide variety of chemical compounds including petroleum derivatives and other inorganic compounds. It is one of the thermal power plants that uses heat energy from petroleum and converts it into electric power.

Hydrocarbons are categorized as either aliphatic or aromatic. Total petroleum hydrocarbons (TPHs) include saturated aliphatic hydrocarbons such as alkanes, alkenes, and cycloalkanes. Out of the hundreds of identified hydrocarbons, 16 compounds are known as polycyclic aromatic hydrocarbons (PAHs) and identified as primary concerns due to their elevated toxicity and harmful effects [4]. Wastewater polluted with

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hydrocarbon has devastating effects on the biota of environment (plants, animals, and humans); these effects include decreased crop yield, lack of dissolved oxygen in water bodies, and effects on marine plants [5].

Hydrocarbons can leach out during irrigation with polluted wastewater, reducing soil fertility, hindering essential nutrients for crop growth, and bringing about a reduction in crop yield and accessible food for households [6]. Another effect is the lack of oxygen where the presence of hydrocarbons in water prevents gas exchange and the penetration of sufficient light through the water column, affecting their utilization by aquatic plants and making them incapable of photosynthesis. This leads to aquatic plant death and can pass the hydrocarbons up to the food chain to consumers including animals and humans [7]. Effluent from power plants poses potential risks to environment because it contains hydrocarbons and heavy metals in high concentrations. For water oil pollution remediation, three approaches can be considered: physical (dissolved air flotation, filtration) [8], chemical (advanced oxidation process, electrochemical) [9] and biological (activated sludge, trickling filter) [5]. These traditional methods are usually expensive, energy-intensive and may sometimes cause additional pollution or generate extra sludge; therefore, new remediation methods that are effective, precise, less expensive and not harmful for the environment should be investigated.

Constructed wetland (CW) is a broad term referring to technologies that use plants and their associated microorganisms (particularly in the rhizosphere), to reduce, remove, or detoxify hazardous pollutants such as heavy metals, pesticides, petroleum hydrocarbons, chlorinated solvents, BTEX (Benzene, toluene, ethylbenzene and xylene) compounds, and excess nutrients in the soil, air, and water [10]. Phytotechnology is defined as the use of efficient biologically selected or potential plants to remove or degrade pollutants, reducing the concentrations and toxic effects of contaminants in the environment [11]. It combines biological activities through synergetic plant-microbe interaction, physical process through medium/substrate filtration with the assistance of plant roots, and finally chemical reactions between root exudates and pollutants. CW system as a green technology achieves cost-effective, valuable, and sustainable technique in treating wastewater [12]. Various plant species namely *Lepironia articulata* [13], *Rhizophora mangle* [14], *Salix smithiana* [15], *Eleocharis ochrostachys* [16], *Scirpus grossus* [17] and *Festuca arundinacea* [18] have been recently utilized to reduce hydrocarbons in contaminated medium.

Constructed wetlands (CWs) are human-made systems developed by the combination of green plants with their associated microorganisms, to mimic role of natural wetlands, and appropriate soil, to improve water quality [12,19]. CWs have been used to treat various point and nonpoint sources of water pollution, including domestic wastewater [20], coal mine drainage [21], agricultural effluent [22], landfill leachate [23], and storm water runoff [24]. CWs have also been applied as a secondary application for domestic wastewater, industrial and agricultural wastewater, tertiary treatment, polishing wastewater, urban runoff, and contaminated groundwater [25]. To our best of knowledge, application of CWs to treat effluent from power plants is still limited and scattered. This green treatment technology is currently of great interest because it achieves high treatment efficiency with reduced operation energy, lower cost, and easy maintenance requirements [26,27]. Although prior work has been done applying hybrid CWs to treat sewage [28], coffee mill effluent [29], and diesel-contaminated wastewater [30], none has reported on hybrid CW application for effluent from power plant. Therefore, in this study, a continuous-flow hybrid system comprising a horizontal subsurface flow followed by a surface flow (HSSF-SF) was installed for the treatment of effluent discharged from the Al-Najibiyia Power Plant. *Phragmites australis* (Cav.) was planted in in HSSF CW system and followed by *Hydrilla verticillata* (L.f.) Royle in SF CW system.

The selection of *P. australis* and *H. verticillata* were based on the phytoremediation potential of oil-diesel-contaminated medium [31,32] and organic compounds [33,34] respectively. This research hypothesizes that the application of plant-assisted remediation using *P. australis* and *H. verticillata* can enhance the degradation of hydrocarbons. The utilization of hybrid CW system with *P. australis* and *H. verticillata* provides innovative knowledge in the remediation of areas impacted by oil activities. Thus, the objective of this study was to assess the performance of hybrid CW planted with *P. australis* and *H. verticillata* in removing hydrocarbons present in the wastewater. We anticipate that treated effluent will be viable for reuse and irrigation purposes, which is a very important issue in the region. The present study also focused on providing a better understanding of the impact of system parameters, including exposure time and presence of plants, on the overall removal efficiency, and determining the concomitant plant responses to various contaminants and conditions.

2. Materials and methods

2.1. Constructed wetlands system design

A hybrid constructed wetland system with a continuous flow rate at 3 L/day with 5-day hydraulic retention time was constructed to treat industrial wastewater discharged from the Al-Najibiyia Power Plant in Basrah City, Iraq. The retention time was setup to 5 days according to [35] who recommended 4 to 15 days. The system (Fig. 1) consisted of three parts, starting with a storage tank, two glass basins (30 cm width × 30 cm depth × 60 cm length), one with a horizontal subsurface flow (HSSF) system with an emergent plant species, *P. australis* (planted with 18 one-month old plants), followed by a glass basin with a surface flow (SF) system planted with a 100 g submergent plant species, *H. verticillata*. Another similar series of two glass basins without plants served as a contaminated control HSSF (CC HSSF) and contaminated control SF (CC SF). Each basin had a front-to-back height difference of about 1 cm to encourage the easy flow of treated wastewater [13,20]. The pilot hybrid constructed wetland was conducted in outdoor environment at the University of Basrah for a 72-day period under natural conditions in January–March 2022 with temperature ranging between 15 and 25 °C.

The HSSF basin was filled with a local substrate to a height of about 20 cm. The substrate consisted of two gravel layers (course, 3 ± 1 cm in diameter, and fine, 1 ± 0.5 cm in diameter) with a height of 11 cm, followed by a layer of fine sand to a height of 9 cm above the top surface of the substrate media. Based on [25] study, the presence of gravel is essential to ensure normal growth of the plants while avoiding asphyxiation conditions of the bottom part of the plants [36,37] and provide anoxic conditions all through the treatment period [15,38,39]. Sand was used to observe the direct toxicity effect of the hydrocarbons since it contains minimum concentrations of nutrients (macro and micro) [40].

According to [41], the volume capacity of all basins can be determined as length (L) × width (W) × Height of substrate (H), which is 36 L (60 cm × 30 cm × 20 cm = 36,000 cm^3). For the basin with HSSF, which has a 20-cm-height substrate with a gravel porosity of about 30 %, the volume capacity is 10.8 L (30 % of 36 L). CWs were planted with *P. australis* in the HSSF basin and *H. verticillata* in the SF basin. The *P. australis* and *H. verticillata* plants weighed 4.4 ± 0.4 g and 1.0 ± 0.02 g per plant, respectively.

2.2. Characteristics of Al-Najibiyia Power Plant effluent and analysis of water quality in the hybrid HSSF-SF system

Wastewater was obtained from the main wastewater drain at Al-Najibiyia Power Plant, located in Basrah City in Iraq. Table 1 illustrates the characteristics of raw wastewater from the Al-Najibiyia Power Plant. It was alkaline at pH 8.17, 4.20 ± 2.38 mg/L dissolved oxygen (DO), 647 ± 66.47 mg/L total suspended solids (TSS), 355 ± 77.78 mg/L chemical oxygen demand (COD), 469 mg/L TPH, and 1547 mg/L PAH. Comparing with the Iraqi regulation limits, all parameters of DO, TSS,

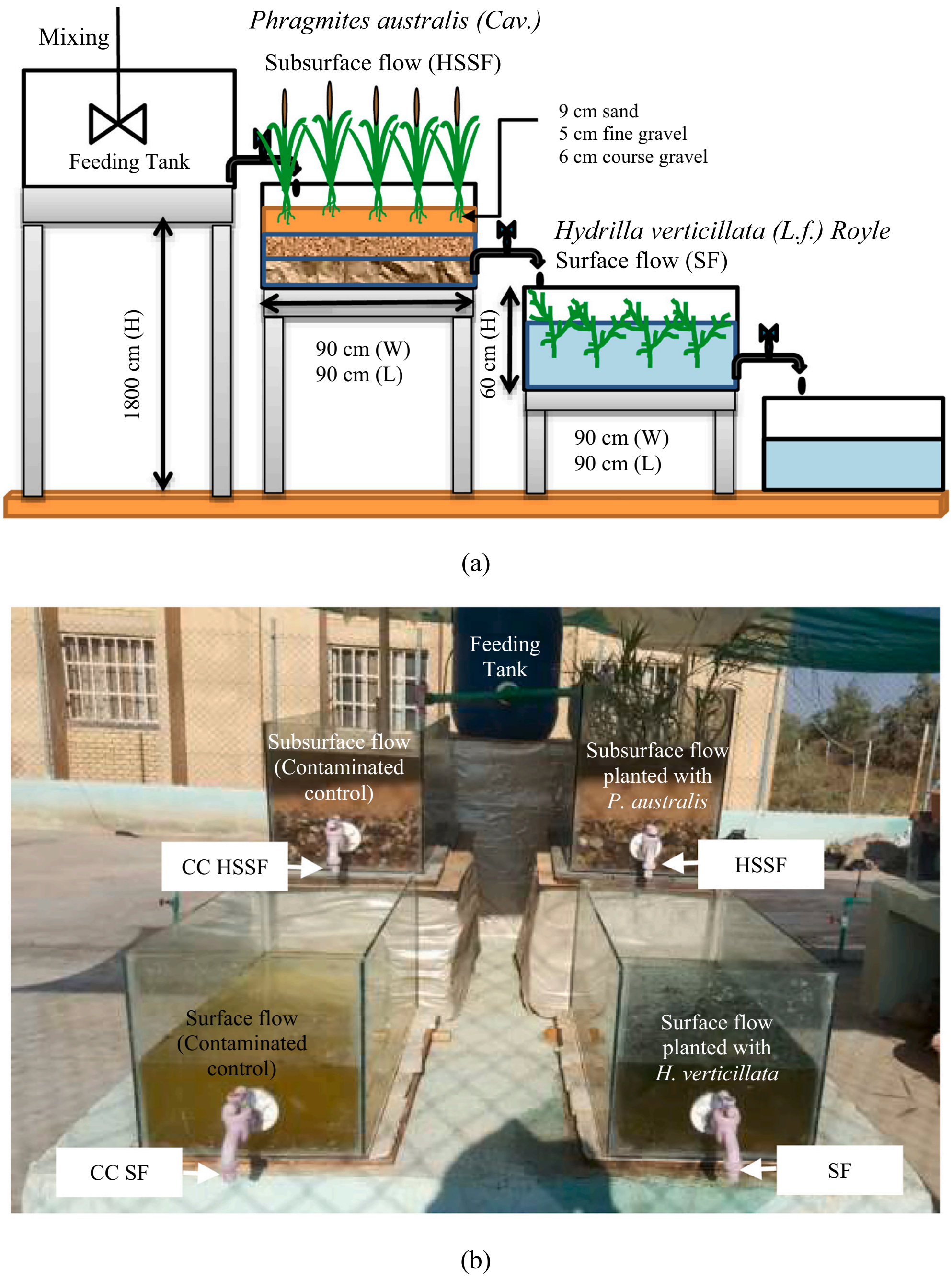

Fig. 1. Design of a hybrid HSSF-SF constructed wetland system, (a) from the side view, and (b) the real set-up photographed from the front view.

COD and TPH are above the regulated values, indicating the requirement for treatment before being discharged to the environment. During the 72-day phytoremediation period, treated water was collected on 0, 7, 14, 28, 42, and 72 days from four sampling points labelled in Fig. 1(b) as CC HSSF, CC SF, HSSF and SF to evaluate the performance of the system. Some measurements were conducted directly after sampling, such as COD, DO, pH, TSS, and temperature. Analyses of COD and TSS were performed following standard methods [42]. All samples were analyzed in the environmental laboratory of the University of Basrah and compared with the environmental quality (industrial effluent) regulations 2011 for wastewater in Iraq [43].

2.3. Plant growth monitoring

The wet and dry weights of *P. australis* and *H. verticillata* were measured during the 72-day phytoremediation period. Two plants each of *P. australis* and *H. verticillata* were harvested every sampling time to estimate the biomass of the plants. The roots and stems of the plants were rinsed with tap water then with distilled water to remove all soil and other particulate matter, dried with a cloth, and weighed to determine the wet weight. All two-plant samples were dried in an oven at 70 °C for 72 h to determine their dry weight [15]. The plant response to the hydrocarbons can be determined by calculating the relative growth rate (RGR) following Eq. (1) [40].

$$\text{Relative growth rate}\ \left(\text{gg}^{-1}\ \text{d}^{-1}\right) = \frac{ln\text{DW}_2 - ln\text{DW}_1}{\text{Exposure period (day)}} \tag{1}$$

In Eq. (1), DW_1 and DW_2 represent the initial and final dry weight of plants, respectively.

Table 1
Characteristics of the raw effluent at Al-Najibiyia Power Plant.

Parameter	Unit	Industrial wastewater (mean ± standard deviation)	Iraq regulation limits for industrial effluent (2011)
Temperature	°C	21.2 ± 3.46	<35
pH	–	8.7 ± 1.2	6–9.5
DO	mg/L	4.2 ± 2.38	>5
TSS	mg/L	647 ± 66.47	60
COD	mg/L	355 ± 77.78	100
TPHs	mg/L	469	10
PAHs	mg/L	1547	–

2.4. Analysis of chlorophyll content

Chlorophyll content was determined using the modified method of [44], where 0.2 g of soft leaves were taken and crushed with ceramic mortar in acetone (with 80 % concentration), and filtered using a centrifuge at 5000 rpm for 5 min. The chlorophyll concentration was estimated by a spectrophotometer (V-1800PC, Mapada, China) at wavelengths of 645 and 663 nm. The total chlorophyll in mg/L was calculated from Eq. (2):

$$\text{Total chlorophyll (mg/L)} = 18.6\ \text{OD (645 nm)} + 12.7\ \text{OD (645 nm)} \tag{2}$$

In Eq. (2), OD is the optical density at the indicated wavelength.

2.5. Hydrocarbon removal from wastewater

Two types of hydrocarbons in water samples, total petroleum hydrocarbons (TPHs) and polycyclic aromatic hydrocarbons (PAHs), were extracted according to [45,46], respectively, and analyzed using gas-liquid chromatography (GC) (7890A, Agilent, United States). Hydrocarbons in water samples were extracted following [45]; 100 mL of sample was mixed with Chloroform (1:1) in a separating funnel and left to settle. The organic phase was easily separated because it was heavier than water; samples were then left alone while the solvent evaporated. The hydrocarbons were dissolved in 25 mL of *n*-hexane and passed through a chromatographic separating column containing glass wool at the bottom, and then silica gel (100–200 mesh), alumina (100–200 mesh), and anhydrous sodium sulfate (Na_2SO_4). The aliphatic fractions were eluted from the column with *n*-hexane (25 mL) for TPHs, whereas the aromatics (PAHs) were eluted with benzene (25 mL). The samples were then air dried and stored until detection of aliphatic and aromatic hydrocarbons by GC. The percentage of hydrocarbons removed in each sampling day was determined from Eq. (3) [39,47].

$$\text{Removal (\%)} = \frac{(\text{H}_0 - \text{H}_t)}{\text{H}_0} \times 100 \tag{3}$$

In Eq. (3), H_0 is the initial hydrocarbon (TPH or PAH) concentration (day 0), and H_t is the hydrocarbon concentration on the sampling day during the 72-day of experiment.

2.6. Statistical analysis

SPSS version 23 (SPSS Inc., United States) was used to analyses results; Analysis of Variance (ANOVA) was used to determine significance level between independent variable (time) and the dependent variables, i.e., physical parameters such as removal efficiency of COD and hydrocarbons, and Chlorophyll content in plants [48]. To minimize experimental error, all the analyses were performed in duplicate. Statistical significance was defined at a confidence level of $p < 0.05$ [40].

3. Results and discussion

3.1. Variation of physical and chemical parameters

All physical and chemical parameters were recorded throughout the experimental period showed better treatment system performance in the systems with plants relative to the control without plants (Table 2). In general, the results showed that the temperature mean values ranged between 14 °C and 19 °C in the tank with plants while it ranged between 15 °C and 21 °C in the control tank without plant throughout the 72 days with no significant differences among system types. This temperature is normal for moderate regions like Basrah City in winter.

The mean pH value was in the range of 6.5–8.5, indicating suitable conditions for removal of pollutants and also appropriate conditions for different water reuse applications. pH was approximately constant in the final discharge and was within the acceptable ranges when compared with the influent, thus verifying the buffering capacity of the CWs [49]. [35] demonstrated that the pH of water and soils in wetland systems has a strong impact on the path of many chemical reactions and physical and biological processes, for example, partitioning of ionized and unionized forms of acids and bases, cation exchange, solid and gas solubility, and biological transformations; moreover, many metabolic activities are pH-dependent, and are less effective if the pH is too high or too low.

Results showed that the hybrid system achieved higher DO levels (8.0 ± 1.0 and 10.0 ± 0.1 mg/L in the HSSF and SF, respectively) compared to those of the control (6.5 ± 1.0 and 7.5 ± 1.0 mg/L in the CC HSSF and CC SF, respectively) after 7 days of the experiment, and compared to the raw wastewater, which had a lower reading (4.2 ± 0.5 mg/L). The difference in dissolved oxygen concentration between with and without plant is due to the availability of plants which improve the biodegradation pollutants [50]. In phytoremediation, plants enhance dissolved oxygen and exudates in the rhizosphere facilitating microorganisms to degrade organic pollutants [51]. Constructed wetlands substrate enhances dissolved oxygen and supports microorganisms in biodegradation and removal efficiency of organic matter and nitrogen [52]. Statistical analysis showed that there were significant differences ($p \leq 0.05$) in DO levels based on system type and time. Furthermore, the presence of plants had an influential role in increasing DO compared to systems without plants: the highest level was 11.8 ± 0.14 mg/L in the SF system after 14 days and the lowest level was 5.25 mg/L in CC HSSF at the end of the experiment.

According to the systems, TSS gradually declined within HSSF until at the end of 72 days the mean value was 52.3 mg/L, which is approximately agreed with the Iraqi standard limits for rivers (2011) which is 50 mg/L. The highest value was recorded in CC HSSF after 7 days of treatment. Higher removal efficiencies of 91.3 % and 88.8 % were recorded during the last week in the hybrid system with plants compared to systems without plants (67.4 % and 68.7 %). The total removal efficiency of the system illustrated the effective role of plants in HSSF and FS systems in removing suspended solids by physical processes

Table 2
Physical and chemical parameter variation of effluent during 7 to 72 days of experimental period.

Parameter	Feeding tank (Day 0)	HSSF	SF	CC HSSF	CC SF
Temperature (°C)	16 ± 2	16 ± 2	18 ± 2	18 ± 2	16 ± 2
pH	7.5 ± 1.0	7.0 ± 1.0	8.0 ± 1.0	8.5 ± 1.0	7.5 ± 1.0
TSS (mg/L)	647 ± 50	137 ± 10	117 ± 10	320 ± 10	210 ± 10
DO (mg/L)	4.2 ± 0.5	8 ± 1	10 ± 1	6.5 ± 0.7	7.5 ± 1
COD (mg/L)	355 ± 77.78	85 ± 8	70 ± 8	233 ± 10	220 ± 7

such as sedimentation and filtration [29].

COD, on the other hand, showed the significant influence of plants and system type, as the final discharge reached 13.30 ± 0.00, 31.95 ± 0.07, 133.1 ± 0.14, and 125.75 ± 0.21 mg/L COD in HSSF, SF, CC HSSF, and CC SF, respectively, removing 96.2, 91, 62.54, and 64.54 % in HSSF, SF, CC HSSF, and CC SF, respectively after 72 days. Conversely, time had no significant effect on the COD level ($p \leq 0.05$). The higher efficiency of systems with plants in removing COD compared to the systems without plant indicates that plants (*P. australis* and *H. verticillata*) were able to provide beds with the sufficient oxygen to support the aerobic degradation of the organic matter in the wastewater [53].

3.2. Monitoring of plant growth

Fig. 2(a) shows *P. australis* and *H. verticillata* observations for day 0 and day 72 days of the wastewater treatment period. It indicates that *P. australis* can survive and grow well while *H. verticillata* grew normally until day 42, and then the growth and biomass decrease gradually.

The results in Fig. 2(b) show the wet and dry weight of plants during the 72-day experiment to determine the physical signs of toxicity reflected by changes in the plant biomass. Initially, the maximum dry weight of *P. australis* was recorded in the first day of treatment before the plants were affected by the contaminated effluent. Note that the minimum dry weight was recorded on day 7 (0.7 ± 0.01 g), when the plants attempted to adapt to the application of contaminated effluent, and additionally, to concurrent cold weather. It can be seen that the plants managed to adapt to the new environment, as the dry weight increased on days 14 to 28 (1.1 ± 0.01 and 5.8 ± 0.1 g, respectively). After 28 days of exposure, there was a gradual reduction in the dry weight of the plants until the end of experiment (1.9 ± 0.03 g). This might be due to the toxic effects of the continuous feeding of contaminated effluent. It has been shown that the presence of hydrocarbons from diesel similarly impacted the growth of *Lepironia articulata* [39], i.e., the plants in a reactor with higher diesel concentration showed lower dry weight compared plants in reactor with lower diesel concentration. Statistical analysis showed that wet weight and dry weight of *P. australis* did not decrease significantly with time ($p \leq 0.05$).

H. verticillata had a maximum dry weight after 42 days (5.2 ± 0.2 g), from a starting value of 0.4 ± 0.1 g before the plant was subjected to the effluent. From days 0 to 42, the dry weight of plants increased, giving evidence that this plant could tolerate the effluent treated by the HSSF system: the concentration hydrocarbons received in the SF system was lower than the hydrocarbon concentration fed into the HSSF system. After 42 days, it can be seen that the plant growth was affected by the effluent, because the dry weight decreased gradually to 2.7 ± 0.2 g on day 72. This might be due to the excessive concentration of hydrocarbons that the plant could tolerate, which became toxic and began inhibiting the growth of the plant. According to [39], plants exposed in high concentration of hydrocarbons will die because the roots stop growing and then rot and become smelly due to insufficient oxygen, which promotes anaerobic degradation processes. Statistical analysis showed that the wet weight of *H. verticillata* did not vary with time, but dry weight was significantly affected by time ($p \leq 0.05$).

In addition, the effect of the contaminants on both plants can be evaluated by looking at the trend of RGR between two consecutive days of exposure. It can be observed that *P. australis* was negatively affected

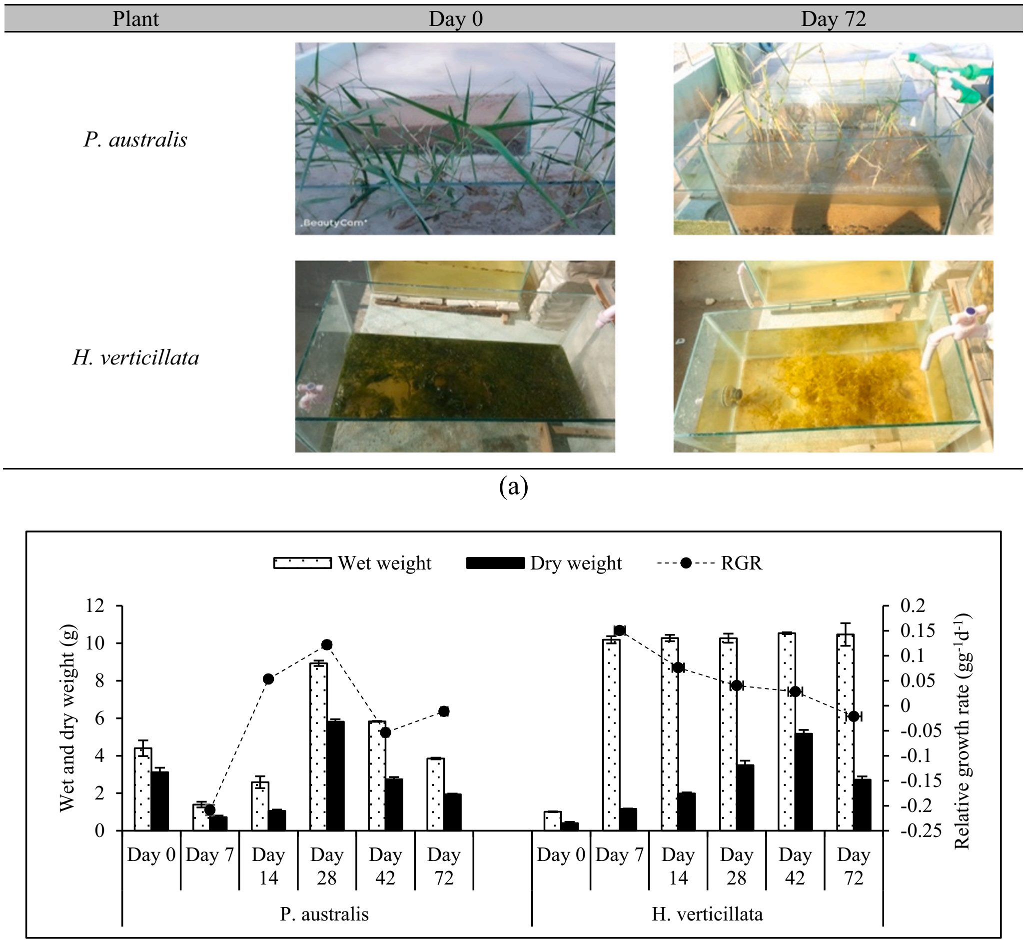

Fig. 2. Plant growth response: (a) physical observation, and (b) wet and dry weight and relative growth rate (RGR) of *P. australis* and *H. verticillata* during the experimental period (72 days) (bars indicate the standard deviation of the data).

by the contaminant during the early stages of experiment (days 0–7), where the RGR values were at the lowest values compared to the other RGR values. According to [54], the initially existing leaf area is low and has a low ratio of photosynthetic tissues to respiration during early exposure. In contrast, the plant showed a positive growth response during days 14 and 28 (increased dry weight), and the RGR values were higher than on other days. The increased RGR was due to faster leaf growth and the maximum number of young tissues involved in photosynthesis [54]. Towards the end of experiment (days 28–42), the RGR decreased due to the aging of the leaves and a relative decrease in photosynthesis [54]. On the other hand, the RGR for *H. verticillata* keeps decreasing until the end of exposure period even though the dry weight increased up to 42 days. This was due to the decrease in the photosynthesis [54] caused by the contaminated water. In another study utilizing sago mill effluent as a source of contaminated water, it was observed that the effluent contributed to positive plant growth when the plants used the nitrogen, phosphorus and potassium existing in sago effluent as essential nutrients [48].

3.3. Chlorophyll content in plants

The results in Fig. 3 illustrate the total chlorophyll content in *P. australis* during the experimental period. It was observed that the highest chlorophyll content in *P. australis* of 22.28 mg/L was recorded after 14 days while the lowest content of 11.1 mg/L was recorded after 28 days (compare to the control plant, 28.5 mg/L) due to the high loading of pollutants discharged to the system at that time. Statistical analysis showed that there were no significant differences in chlorophyll content between the treatment plant and the control plant as a function of experimental time ($p \leq 0.05$).

The total chlorophyll content was also decreased in *P. australis* at the end of the 72 days to 13.13 ± 0.1 mg/L compared to 18.9 ± 0.2 in the control plant. Moreover, the chlorophyll content in *P. australis* generally was higher than *H. verticillata*, which may indicate the expected higher resistance of this plant to the stress of pollution [55]. There was also a significant reduction in total chlorophyll content in *H. verticillata* during the experimental period, reaching 0.14 ± 0.3 mg/L at the end of the 72 days, compared to the first day with 15.62 ± 0.11 mg/L, while the control plant had 8.3 ± 0.13 mg/L on the same day (Fig. 3). Statistical analysis showed that there was a significant decrease in chlorophyll content for the treatment plant compared to the control plant as a function of experimental time ($p \leq 0.05$). After the 28 days, *H. verticillata* had changed in colour (from healthy green to yellowish green), showed some leaf abscission, and showed a lack of chlorophyll as indicated by chlorosis symptoms [56]. This may occur due to the increase of hydrocarbons inside the plant and accumulated in the leaves. The reduction in chlorophyll may be stimulated by several factors such as aggressive environmental pressures [57].

A hybrid reed-bed CW with aeration was employed for treating domestic wastewater using *Scirpus grossus* with removals of 84.7 and 71.0 % for NH_4-N and PO_4-P, respectively [28]. [54] observed a sharp decline in chlorophyll in *Amorpha fruticosa* affected by different petroleum-contaminated soil concentrations in the range 5–20 g/kg. In another study [56], a hybrid system comprising two units of a vertical flow (VF) wetland followed by a floating treatment (FT) wetland was tested. Removal percentage was higher in the FT wetland with higher biological oxygen demand (BOD) (56.2 %) and COD (58.4 %) when compared with the SF wetland (BOD of 52.5 % and COD of 48.1 %).

3.4. Hydrocarbon removal from wastewater

3.4.1. Total petroleum hydrocarbons (TPHs)

The *n*-alkanes in the raw industrial wastewater sample before treatment contained carbon atoms ranged from C_8 to C_{30} in addition to pristine ($C_{19}H_{40}$) and phytane ($C_{20}H_{42}$). During the experimental period, C_9-C_{15} species of *n*-alkanes were not detected in water samples because they easily evaporate and are decomposed by microorganisms, while C_{28}-hC_{30} *n*-alkanes have the ability to settle down [58]. The average TPH in raw wastewater was 469.0 ± 109.4 mg/L, but after seven days in the treatment system, it decreased to 28.80 ± 2.10 and 22.0 ± 1.02 mg/L in the HSSF and SF systems, respectively, and 67.6 ± 1.90 and 55.1 ± 0.50 mg/L in the CC HSSF and CC SF systems, respectively.

The hybrid system recorded a clear increase in *n*-alkane concentrations after 28 days of the experiment, due to their high concentrations in the feeding wastewater during that period; the lowest total value was at the end of the 72 days when it reached 5.41 ± 2.00, 8.5 ± 2.00, 31.92 ± 5.00, and 29.12 ± 3.00 mg/L in the HSSF, SF, CC HSSF, and CC SF systems, respectively. There were significant differences in *n*-alkanes concentrations with different system types and experimental times ($p \leq 0.05$). Fig. S1 presents the GC analysis of *n*-alkanes for the untreated power plant effluent (0 day) and the treated water after 72 days of the experiment. The removal of *n*-alkanes reached a maximum of 97.75 and 95.3 % in the HSSF and SF systems followed by the contaminated control systems (CC HSSF and CC SF) which were 78.8 and 71.6 %, respectively, at the end of the 72 days. The high percentage of TPH degradation by plants might be due to the microbial assistance through biodegradation.

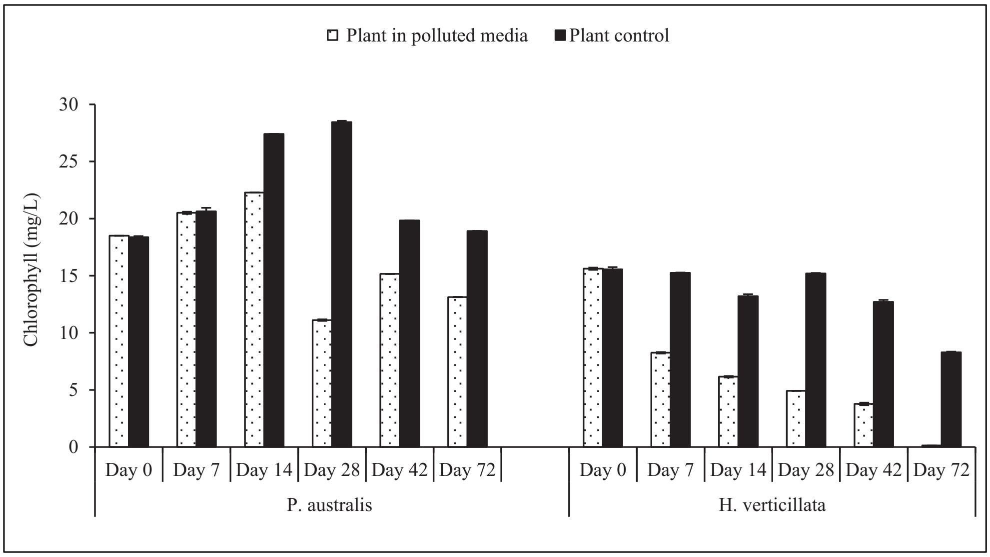

Fig. 3. Chlorophyll content of *P. australis* and *H. verticillata* during the experimental period (72 days) (bars indicate the standard deviation of the data).

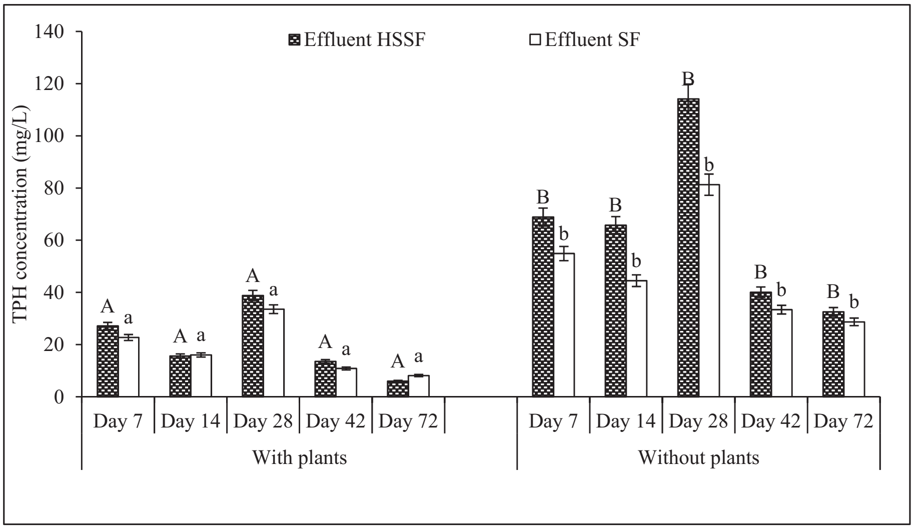

Fig. 4. Concentration of TPHs in systems with and without plants during the experimental period (bars indicate the standard deviation of the data). Letters (A and B; a and b) represent statistically significant differences on a specific day when compared with plant and without plant for effluent HSSF and effluent SF respectively.

Fig. 4 shows *n*-alkane concentrations for different system types over time. The lowest *n*-alkane concentration was recorded in SF followed by HSSF at the end of 72 days.

3.4.2. Polycyclic aromatic hydrocarbons (PAHs) in water

The industrial wastewater sample had high PAH content before treatment, reaching 1636.48 ± 125.62 mg/L. As with previous results for *n*-alkanes, clear increase in PHA was recorded after 28 days of the experiment due to the high value of PAH in the untreated wastewater during that period; the lowest total value was on day 72. Statistical analysis showed that there were significant differences in PAH concentrations based on system type and on time ($p \leq 0.05$).

The lightest PAHs with recorded values during the experiment were acenaphthene, fluorene, phenanthrene, and anthracene, while heavy PAHs were dominant for all system types, i.e., benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, indeno[1,2,3-cd] pyrene, dibenz[*a*,*h*]anthracene, and benzo[*g*,*h*,*i*]perylene were detected at higher levels during the experiment in all system types. The difference in impact between vegetated and non-vegetated treatments, concerning the removal of PAH, was substantial. The removal of PAH at the end of 72 days reached 96.8 and 96.2 % in HSSF and SF compared to 51. 9 and 56.8 % in CC HSSF and CC SF. Fig. S2 shows the GC analysis for the untreated industrial wastewater and the treated water after 72 days of the treatment.

Fig. 5 shows the residual concentration of PAH in systems with plants compared to systems without plants during the experimental period (72 days). Table 3 summarizes contaminant removal by hybrid systems (the main aim), showing significant differences between planted and unplanted CWs ($p < 0.05$). The hydrocarbon removal efficiency was 90–96 % and 50–78 % for CWs with and without plants, respectively. Higher hydrocarbon removal might be due to the presence of bacteria in the rhizosphere [59]. Jain et al. [12] have recently proved that the

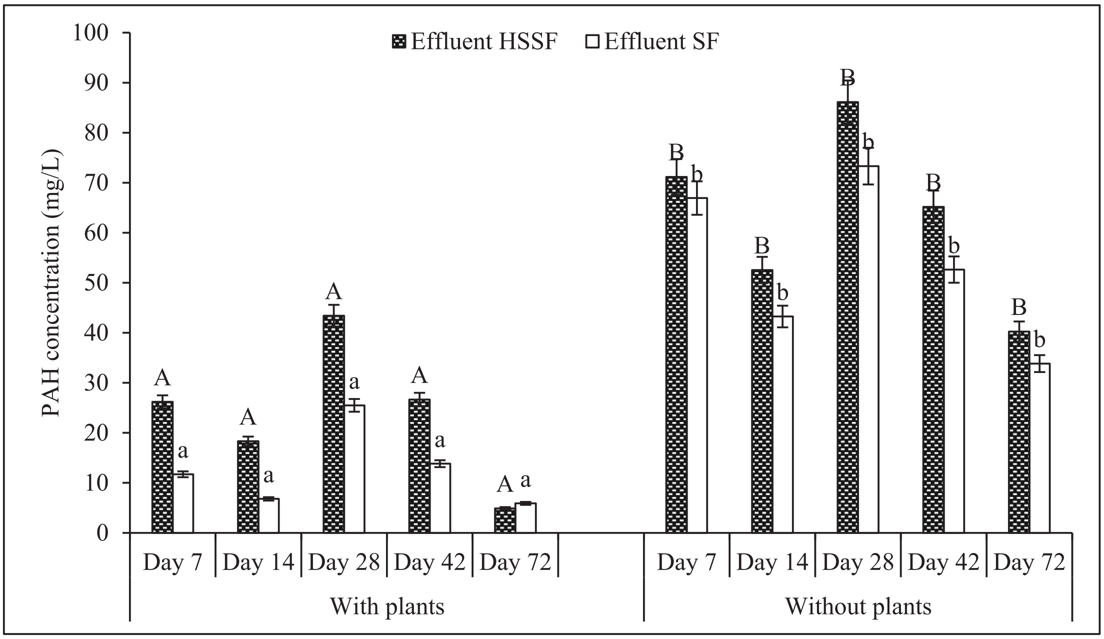

Fig. 5. Concentration of PAHs in systems with and without plants during the experimental period (bars indicate the standard deviation of the data). Letters (A and B; a and b) represent statistically significant differences on a specific day when compared with plant and without plant for effluent HSSF and effluent SF respectively.

Table 3
Difference in quality of treated water in the hybrid system with and without plants after 72 days.

Parameter	Unit	Initial concentration in feed tank	System with plants: mean (removal %)			System without plants: mean (removal %)		
			HSSF	SF	Hybrid system	CC HSSF	CC SF	Hybrid system
TSS	mg/L	647	52.3 (91.9 %)	66.5 (−28.1 %)	66.5 (89.7 %)	196 (69.7 %)	187 (4.1 %)	187 (71.0 %)
COD	mg/L	355	13.3 (96.3 %)	32.0 (−140.2 %)	32.0 (91.0 %)	133 (62.5 %)	126 (5.5 %)	126 (64.5 %)
TPH	mg/L	469	5.41 (98.8 %)	8.5 (−35.0 %)	8.5 (98.2 %)	31.9 (93.2 %)	29.1 (−13.4 %)	29.1 (93.8 %)
PAH	mg/L	1547	4.63 (99.7 %)	5.88 (−20.1 %)	5.88 (99.6 %)	37.8 (97.4 %)	31.6 (−18.9 %)	31.6 (97.8 %)

rhizobacteria from *Lepironia articulata* helps in the degradation of PAHs. Fahid et al. [31] used floating treatment wetlands to remediate diesel-contaminated water and after 90 days of treatment, the maximum reductions were 95.8, 98.6, 97.7, 95.2, and 98.9 % for hydrocarbons, COD, BOD, total organic carbon, and phenol, respectively. The results from this study are in agreement with [15], in which PAH removal percentages from water with *Eleocharis ochrostachys* were 89.1, 91.3, 73.0 and 71.6 % at various diesel concentrations of 0.5, 1, 2 and 3 % v/v, respectively.

3.5. *Inclusive performance of hybrid HSSF-SF system*

When applying the hybrid constructed wetlands system for treating industrial wastewater polluted with hydrocarbons is a highly-effective solution with considerable short time (Fig. S3). The hybrid system with two plants of *P. australis* and *H. verticillata* have showed the ability to increase hydrocarbons degradation through plant metabolism while *P. australis* showed better growth characteristics than *H. verticillata* which reflects the high resistance of this plant to the pollutants. The presence of a substrate in HSSF has had a significant effect on pollutants removal, especially TSS and COD since it acts as a filtration medium. As the individual in SF systems, reduction in TSS, COD, TPH, and PAH were not efficient in the system as the concentration increased at end period of treatment, only significant removed in the HSSF. As shown in Table 3, removal efficiency of TPH and PAH were 98.8 and 99.7 % in HSSF as compared to SF system which got negative removal efficiency due to plant effective by hydrocarbons. Meanwhile comparing unplanted systems, in two systems SF and HSSF removals were positive due to not availability of plants and was not significant ($p > 0.05$) for all sampling days. The plant in system the HSSF was able to remove the contamination but the presence of phytoremediation plants helps to achieve a greater level of removal more rapidly while in system SF phytoremediation and removal percentage decline in the plant ability with reduce treatment because plant damaged or accumulation of pollutants in plants release to the water after plant died this is phenomenon agreed with the [60].

4. Conclusions

This study proposes the utilization of hybrid CW systems for the treatment of hydrocarbon-contaminated wastewater. Results after 72 days of exposure of industry wastewater to the continuous-flow hybrid system exposure indicated that *P. australis* and *H. verticillata* could survive and tolerate the contaminated wastewater containing hydrocarbons. The hybrid system achieved significant reductions in TPHs and PAHs. The removal efficiencies were 95.0 ± 2 % and 96.0 ± 2 % (TPHs); and 71.0 ± 5 % and 51.0 ± 5 % (PAHs) in CWs with plants and without plants, respectively. CW with plants significantly removed TPHs and PAHs from the contaminated effluent better than CW without the presence of plants. Laboratory-scale experiments are very valuable in determining optimal operating conditions for upcoming pilot- and field-scale studies. It is also recommended that future studies to include the study related with microorganism. The results of the plant-assisted treatment process demonstrated enhanced hydrocarbon degradation, implying that the phytoremediation is an effective green bioremediation technology. The findings of this study will shed light on future green remediation technology, not only for power plant but also for industry having effluent contaminated with hydrocarbons to increase the quality of treated effluent.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The data that has been used is confidential.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jwpe.2023.104372.

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