



USE OF VERTICAL SUBSURFACE FLOW CONSTRUCTED WETLAND FOR RECLAMATION OF WASTEWATER CONTAMINATED WITH CONGO RED DYE

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Abstract

The release of untreated dye wastewater into streams is unacceptable not only for aesthetic reasons and its negative effects on aquatic life but also because many dyes are toxic and carcinogenic to humans. Biological treatment methods such as constructed wetlands are more cost-effective and environmentally friendly than traditional techniques. The ability of vertical subsurface flow constructed wetlands units for the treatment of simulated wastewater contaminated with Congo red dye has been studied. The units were packed with Iraqi sand bed that unplanted and planted with *Phragmites australis* or *Typha domingensis* which considered the familiar plants in the Iraqi environment. The efficacy of these units was evaluated by monitoring the pH, DO, Temperature, COD and Congo red dye concentration in the effluents under the variation of detention time (1-5 day) and dye concentration (10-40 mg/L). The maximum reductions for dye and COD were equal to 99.34 and 86.0% respectively for 10 mg/L of Congo red dye after five-day hydraulic contact time. Results proved that the removal of dye concentration and COD increased dramatically with increase of contact time and decrease of dye concentration. The values of monitored parameters adopted to evaluate the wastewater quality (*i.e.* pH, DO, Temperature, COD and Congo red dye) are satisfied the requirements of irrigation water. Based on the FT-IR analysis, functional groups like SiO₂ have significant role in the sorption of dye on the sand beds of used CW units.

Key words: Constructed wetland, *Phragmites australis*, *Typha domingensis*, Congo red dye.

Introduction

The textile industry represents the main source for pollution of water bodies especially the estimates of “World Bank” proved that approximately twenty percent of global water contamination results from the textiles dyeing and treatment. The dyeing steps require to utilize different reagents which are rich with chemical compounds; so, these industrial processes can consider unfriendly with environment (Robinson *et al.*, 2001). The effluents of the textile industry are characterized by presence of clear color which can recognize through visualization and this color forms great problem due to generate huge quantities of wastewater from dyeing and finishing processes. The colored wastewater is heavily contaminated with textile auxiliaries and dyes; so, the production technology and applied chemicals will specify the properties of produced effluents (Sultana 2014). However, the mentioned wastewater is comprised various types of pollutants like inorganic, organic, elemental and

polymeric products (Kant 2012).

Congo red is selected in this work to be the target contaminant because this dye considers weighty sinner when released to the environment. It causes anaphylactic shocks in humans and many previous studies proved that this dye is carcinogenic. In addition, Congo red has the same effect of the other dyes when they discharged in water bodies without being treated properly through disturbs the life cycle of aquatic animals and plants by obstructing the penetration of sunlight.

The dyes treatment is accompanied with many limitations such as the dyes are non-biodegradable, stable to light and oxidation, and can't be remediated by conventional techniques. Alternatively, the approach is shifting towards the use of eco-friendly biological methods to treat wastewater containing dyes. Constructed wetlands (CWs) are engineered systems applied in the last decades due to their suitability, mechanical simplicity, low energy requirement, environmental friendliness and

low cost of operation. Based on the previous studies like Knight (1997), Fonder and Headley (2010) and Vymazal (2010a, 2014), CWs can be classified according to the; 1) water level on the bed, 2) type of the plants and 3) direction of the water flow. Free water surface flow (FWSF CWs) and subsurface flow (SSF CWs) are the main types of CWs based on the water level; however, these wetlands can be classified into the horizontal flow (HF CWs) and vertical flow (VF CWs) based on the direction of water flow (Vymazal, 2013). *Phragmites australis*, *Typhonium flagelliform*, *Eichhornia crassipes*, *Azolla caroliniana*, *Typha*, and *Lemnaete* are popular aquatic plants used in the CWs for removing of dye and other pollutants from wastewater (Azeez and Sabbar, 2012).

The 20 glass basins as VF CWs were situated in Turkey to reduce dye concentration (BB41; 11 mg/L influent) using river sand and *P. australis*, Manchurian wild rice with detention time of 3, 6, 9 and 18 days. The results elucidated that the percentage of dye reduction in 9 and 18-day detention time was higher than those at other times (Keskinan and Lugal Goksu, 2007). The VF CWs were packed by gravel and sand, and planted with *P. australis*. Reactive Black 5, Disperse Yellow 211 and Vat Yellow 46 – polluted water are types of wastewater prepared artificially to treat by mentioned units. Removal efficiencies were found of 90, 84, 93 and 33% for color, COD, TSS and $\text{NH}_4\text{-N}$ respectively at various hydraulic loads through period of five months (Bulc and Ojtrsek, 2008). Also, the VF CW to treat wastewater polluted with Acid Yellow 2G E107 was achieved. Three filter vertical wetlands have been packed with sand, fine gravel and zeolite. One of these units was used as unplanted and the remaining have planted with *Cannaidica* and *Typha angustifolia*. The results signified that the removal percentages of color were 87% for unplanted unit and 98% for others (Yalcuk and Dogdu, 2014). The VF CW for treating of the wastewater contained fertilizer mixed with tap water and textile dyes like direct orange 46, AB113, reactive blue 198 (RB198), and basic red 46 (BR46) at low (7 mg/L) and high (215 mg/L) concentrations was operated. Results revealed that this wetland can remove the BR46 and AB113 dyes with efficiencies ranged from 68 to 96% (Hussein and Scholz, 2017).

The present study aims to study the efficacy of vertical subsurface flow constructed wetlands (VSSF CWs) in the reclamation of simulated wastewater contaminated with Congo red dye. This can be achieved by evaluation the ability of sand planted with *Phragmites australis* and *Typha domingensis* in the elimination of

the Congo red from wastewater based on the effluent dye concentration, COD, dissolved oxygen (DO) in comparison with unplanted beds.

Materials and Methods

Wetland set-up

This work was carried out between 1 September 2019 and 1 April 2020. An experimental constructed wetland system treating the simulated wastewater contaminated with Congo red dye was manufactured and operated under natural environmental conditions (average temperature of 23.25°C) within the location of coordinates (33 18' 18" N 44 30' 19" E) situated on the eastern of the Baghdad, Iraq. The experimental work was designed to assess the system performance by simulating the processes occurring within pilot-scale constructed wetlands. The system included 4 VSSF CW units, allowing wastewater to drain vertically, enhancing aerobically biodegradation of organic matter (Fuchs, 2009). The effects of many operational parameters such as contact time, dye concentration, and type of plant on the performance of these units in the reclamation of contaminated water were evaluated.

Congo red is an acid dye with molecular weight of 696.66 g/mol. It gives a red colour for aqueous solution with $\text{pH} > 5$ and this colour becomes blue at more acidic pH. The concentration of this dye can be measured at wavelength corresponding of maximum absorbance (γ_{max}) equal to 498 nm (Gupta, 2015). This dye was prepared with four different concentrations specifically 10, 20, 30, and 40 mg/L; however, these values are consistent with dye concentrations in the textile wastewater as mentioned by Lavanya *et al.*, (2014).

Identical plastic containers were utilized to represent the units of VSSF CWS which their shape can be explained in Fig. 1. Four containers were designed according to the following dimensions; the total height of 60 cm with top and bottom diameters equal to 50 and 40 cm respectively. Two different layers of gravel were used as a filter media where the coarser gravel with size $> 10\text{mm}$ will form the bottom layer to prevent the outlet clogging and the smaller gravel with size $< 10\text{ mm}$ must situate on the top of the previous layer. The gravel with mentioned distribution is commonly available and suitable for constructed wetlands (Crites *et al.*, 2014). All CW units have the same size and shape with total volume capacity around 90 litres. The outlet valve was located at the bottom of each unit with height of 5 cm above the base. This valve can be used for sampling and empty of treated water; however, it is connected with PVC pipe (12.5 mm in diameter) to maintain the level of water and

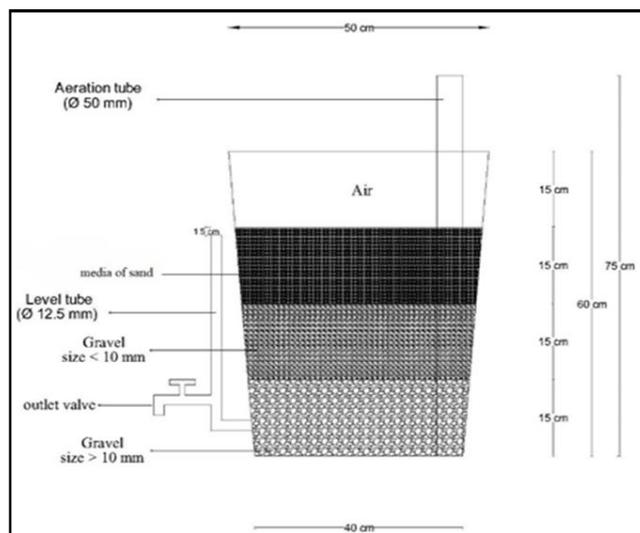


Fig. 1: Details of the pilot-scale unit used in the vertical sub-surface flow CWs for treatment of wastewater.

wastewater within CW units. To ensure the suitable aeration for the filter bed, each CW unit was supplied with perforated PVC tube (50 mm in diameter, 75 cm in length) extended from the above saturated layer until the coarser gravel layer (Stefanakis and Tsihrintzis, 2012). The main bed in the four units is the sand (symbolled by S) with depth of 150 mm that packed above the gravel layers.

Phragmites australis and *Typha domingensis* are the plants selected in this work; however, one unit vegetate with first plant while second plant was inserted in the other unit. The remaining two units was not vegetated and they can be used as control units. The planted units can be recognized from unplanted by adding the symbol “P” with first letter of adopted plant. The first unit will be unplanted and operated with tap water only; so, the designation “CWSC” can be used to recognize it as control unit. The water contaminated with soluble Congo red dye will fed to the remaining three units. The designations for describing of these units will be CWS, CWSPP and CWSPT to facilitate the discussion of measured results.

Vegetation

P. australis and *Typha domingensis* are available in the environment of Iraq and, therefore, they can be used in constructed wetlands. These plants are submerged in the pot and flooded with tap water for two days. Initially, the height of the plants nearly equal to 0.3 m and, to ensure the rapid growth, plants were trimmed to a height of 0.15 m, then planted in the constructed wetland units (Stefanakis *et al.*, 2014) with a density of 5 plants/unit. The plants were irrigated by tap water during October and November while the first of the December

represent the initiation of the experimental operation to evaluate the performance of constructed wetland units in the treatment process. Observations signified that the plants densities are ranged from 18 to 59 plants/unit and their heights not less than 1.1 m for all CWs units after 6 months from plantation process. It seems that some parts of *Phragmites australis* and *Typha domingensis* were suffered from yellowing which may be due to the decreasing of temperature to less than 5°C during the winter season.

Operation of the system

The wetland system was operated in batch flow mode to avoid expenditures such as pumping and automatic control costs. Wastewater was poured directly into the must be stayed within the treatment unit for certain contact time. The treated wastewater was discharged from the wetland unit through “outlet valve” mentioned in Fig. 1. The wetland unit must be remained empty from the wastewater for certain period between any two successive tests and this period called the “resting time”. The whole cycle adopted in the operation of the VF CW system in this work was equal to 5 days. This means that the wastewater pumped for the first day of the operation cycle with closing the outlet valve and keeping the water for 5 days. The sample must be withdrawn from any unit after the end of each day by opening the outlet valve and take approximately 30 mL. After finishing of operation cycle, the outlet valve was opened and treated water can leave the CW along the day; finally, resting of the system extends for ten days to obtain partially saturated conditions. As bed drains, the air is drawn into the bed and re-aerating the microbial.

Measurement of Parameters

The measured parameters include the dye concentration, dissolved oxygen (DO), pH, temperature and chemical oxygen demand (COD, mg/L). The UV-VIS spectrophotometer was used to determine the dye concentration after the filtration of samples using 0.45 µm filter. The DO (mg/L) and temperature (°C) of water were measured using a hand-held mi 605 portable Dissolved Oxygen, MARTINI (Italy). The pH of all samples was measured by using a hand-held E-1 portable the pH electrode Digital Meter (Chine). COD was measured by “closed reflux 5220 C method” described in Standard Methods for the Examination of Water and Wastewater and Environmental Chemistry, Selected Analytical Methods, UNESCO-IHE, (Kruis, 2007).

Results and Discussion

Variation of pH, DO and temperature

The pH is a vital parameter in the identification of water quality and all measurements certified that the pH of treated effluent within the range (6.5-8.5) specified by the World Health Organization (WHO, 2004). For all Congo red dye concentrations (10, 20, 30 and 40) mg/L adopted in this work, the pH was slightly increased in comparison with pH of influent especially at the initial times of treatment process within the range (0.2-0.7) in the CWS, CWSPP and CWSPT units. This slight increase may be due to the interaction between the biofilm and substrate as well as the presence of the plant (Prochaska *et al.*, 2007). Another causes for this increase may be the use of carbon dioxide by plants (Kadlec and Wallace, 2009) as well as the process of denitrification which liberates hydroxyl radicals.

Within the CWs, DO is necessary for the aerobic respiration of microorganisms and it regulates the oxidation in the wastewater (Boyd, 2000). Previous studies elucidated that there are many pathways for oxygen transportation in CWs like roots of macrophytes, contact at the biofilm and atmospheric interface, and oxygen associated with the incoming wastewater (Hou *et al.*, 2016). Under the adopted values of dye concentration, Fig. 2 illustrates that the measured values of DO in the prepared simulated wastewater were varied from 7.45

to 7.9 mg/L due to change of initial dye concentration from 10 to 40 mg/L; however, this variation is expected due to the quality of water used in the preparation and may not relate with dye concentration. It is obvious that the values of DO were decreased with the increase of detention time because the use of this oxygen in the oxidation of organic contaminant present in the wastewater. So, these values were ranged from 4.73 to 6.38 mg/L after 5 days for dye concentrations and units under consideration. It is evident from Fig. 2 that the type of the plant in the CWSPP and CWSPT units is not have significant influence on the concentration of oxygen dissolved in the water. Conversely, DO in the planted units was less than in the unplanted ones with the time because more concentrations of organic material (measured in COD) can oxidize and, consequently, more oxygen will consume. Slight changes in the water temperature can be recognized in the range varied from 17 to 19.2°C for all CWs units under different values of dye concentration. It seems that the measured temperature in each unit is greater than the value of temperature for influent water and this proportional inversely with the values of DO. These increments in the values of temperature may be resulted from the activity of microbial community which supports the

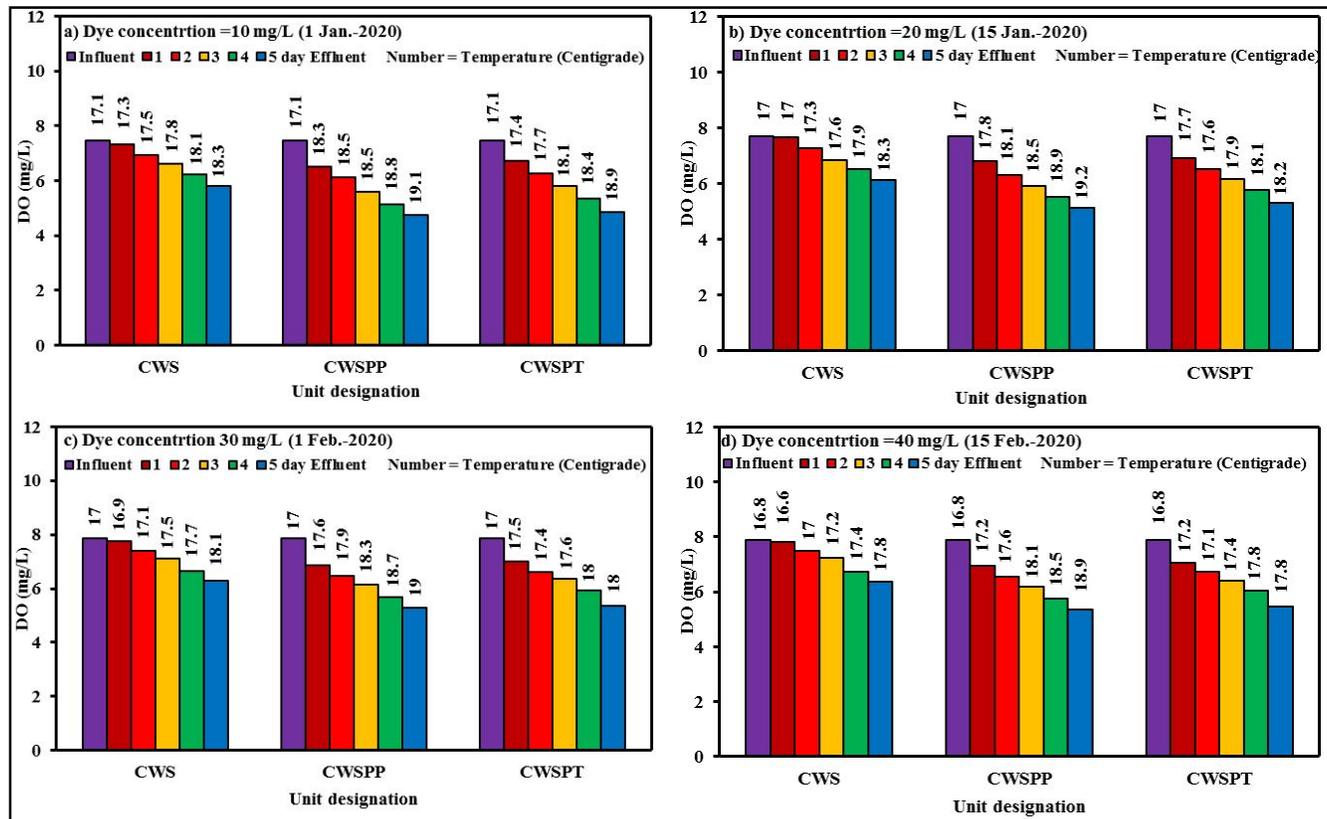


Fig. 2: Influent and effluent concentrations of DO with water temperature versus the detention time in the unplanted and planted CW units packed with sand for different Congo red dye concentrations.

oxidation of contaminants and, consequently, leads to improvement of the water quality.

Removal of organic contaminants

Chemical oxygen demand (COD) was found to be the most commonly used alternative test to BOD for expressing the concentration of organic matter in wastewater samples. The COD analysis has an advantage over the BOD analysis due to lesser time consumption. Initial values of COD for the untreated samples were 30.51, 70.34, 102.33 and 126.2 mg/L corresponding to the dye concentrations of 10, 20, 30 and 40 mg/L respectively. Remarkable reductions in the concentrations of COD were recorded in the vegetated and non-vegetated units that packed with the sand bed Fig. 3. Mostly, the presence of the biofilms within the bed of CW will enhance the oxidation (*i.e.* biodegradation) of organic contaminants; so, this will improve the treatment process and, consequently, the quality of the treated water.

The results certified that the presence of the plants will increase the reduction of COD in mentioned units for adopted concentrations because the plants will provide the required environment for growth of the decomposing microbes within the root zone through increase of oxygen transfer (Taylor *et al.*, 2011). For dye concentration of

10 mg/L, the final removal efficiencies of COD after 5 days in the CWSPP and CWSPT (*i.e.* planted units) have values equal to 84.2 and 86% respectively in comparison with removal in the CWS (*i.e.* unplanted unit) that reach to 56.5%. The increase of the dye concentration will cause a clear decrease in the removal efficiencies which they equal to 48.7, 78.2 and 79% in the CWS, CWSPP and CWSPT units respectively beyond 5 days for 40 mg/L dye concentration. The same figure demonstrated that the increase of the detention time from one day to five days will lead to significant increase in the removal efficiency of COD because the sufficient detention time for wastewater that providing paramount interaction between wastewater and microorganism. For example, the removal efficiencies of COD beyond 1, 2, 3, 4 and 5-day changed within the ranges of (47.4-56.5%), (76.2-84.2%) and (76.7-86%) in the CWS, CWSPP and CWSPT units respectively for 10 mg/L dye and these percentages were decreased with increase of dye concentration. It is clear that the achieved removal efficiencies of COD in the unit planted with *Phragmites australis* (*i.e.* CWSPP) approaches from efficiencies of the one planted with *Typha domingensis* (*i.e.* CWSPT). Finally, all COD concentrations in the treated water produced from units packed with the sand are acceptable in comparison with European Union (EU) prescribed limit for surface water

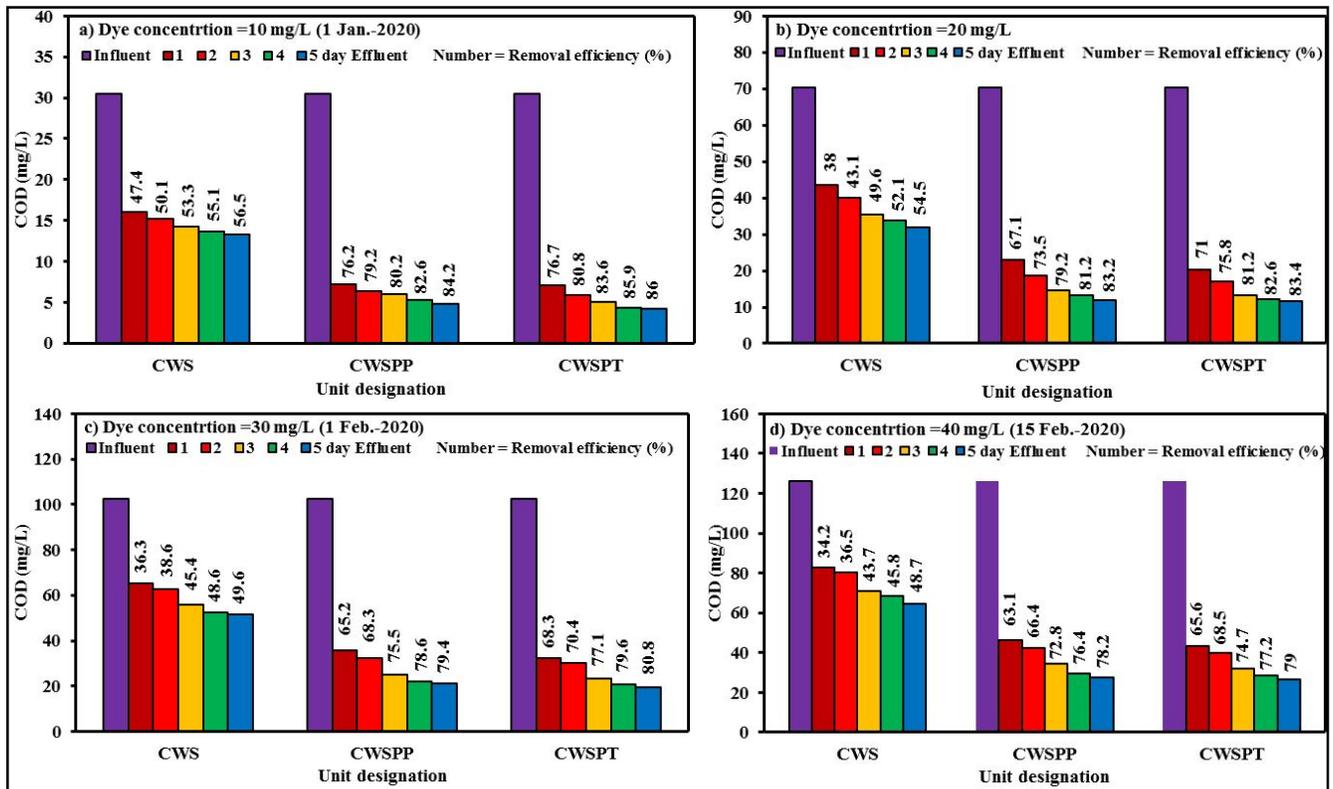


Fig. 3: Influent and effluent concentrations of COD with removal efficiencies versus the detention time in the unplanted and planted CW units packed with sand for different Congo red dye concentrations.

courses that equal to 125 mg/L.

Removal of dye concentration

The degradation of dyes occurs in both aerobic and anaerobic conditions through various processes that include enzymes and/or chemical reduction (Saratale *et al.*, 2011). The color considers the familiar property for wastewater resulted from textile industry, it absorbs and reflects sunlight entering the water, thus interfering with the growth of aquatic species and impeding the photosynthesis (Yadav *et al.*, 2012). The Congo red dye was tested at four different concentrations (target concentrations of 10, 20, 30 and 40 mg/L) to simulate the most concentrations available in the wastewater for different contact times (1, 2, 3, 4 and 5 days) to assess their influence on the performance of VF CWs. Fig. 4 plots the variation of dye concentrations in the treated wastewater as a function of the unit type (vegetated and non-vegetated), type of plant (*Phragmites australis* and *Typha domingensis*), influent dye concentration and detention time. It is evident that all units have high ability for reduction of dye concentration where the effluent concentration not greater than 2.76 mg/L with corresponding reduction efficiency not less than 93.1% at contact time of 5 days regardless the dye concentration entering the CW unit. The lowest value of reduction

efficiency was equal to 89% for unplanted unit (CWS) after contact time of 1 day when the dye concentration of 40 mg/L. Results proved that the efficacy of the CW unit in the treatment of dye concentration will improve with slight values due to increase of contact time, decrease of influent dye concentration and presence of the vegetation. This means that the removal process depends mainly on the packed bed material and biofilms on the solid particles which support the dye degradation as in the COD measurements; however, the used plants have approximately the same effects on the removal process.

Control wetland unit

Additional CW unit packed with sand material and fed by tap water only designated as CWSC was achieved in order to be a reference for performance of CWS, CWSPP, and CWSPT units. The CWSC unit is very important for specifying the effect of red dye addition on the properties of the tap water used to simulate the wastewater contaminated with this dye that identical to the effluents of textile industry. Results signified that the pH of influent tap water to CWSC was equal to 7.3 and it is decreased with the time until stabilized on the 7 after 5 days because the dissolution of carbon dioxide. The pH values of influent tap water and wastewater contained

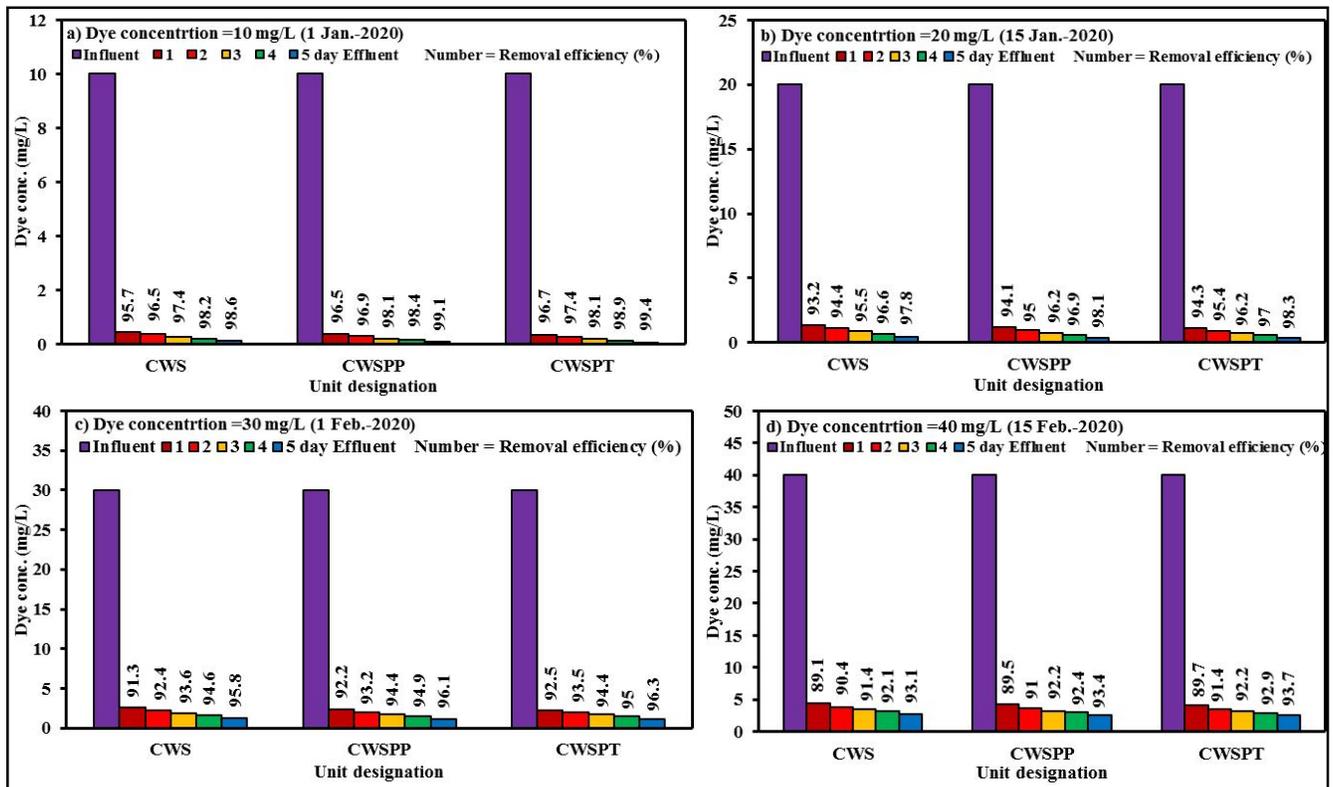


Fig. 4: Influent and effluent concentrations of Congo red dye with removal efficiencies versus the detention time in the unplanted and planted CW units packed with sand.

the red dye are closer to each other.

The monitoring process elucidates that the quantity of dissolved oxygen in the influent tap water for tests of CWSC unit conducted in March is equal to 7.26 mg/L which slightly different from simulated wastewater. This difference may be resulted from the changing in the quality of tap water with the January, February and March months that adopted to implement the mentioned tests on the units packed with sand material. However, a high difference in the influent values of organic material (*i.e.* COD) can recognize between units feeding with wastewater polluted by red dye, (the values equal to 30.51, 70.34, 102.33 and 126.2 mg/L for dye concentrations of 10, 20, 30 and 40 mg/L respectively), and unit irrigated by tap water only with COD of 7.21 mg/L. This high difference in the values of COD is attributed to the addition of organic compound (*i.e.* Congo red dye) in the tap water and, consequently, this water cannot be the source for organic material. Measurements proved that the DO was decreased with the detention time because it exhausted in the oxidation of organic matter. The values of DO and COD in the effluents from CWSC unit beyond 5 days were equal to 5.82 and 3.05 mg/L respectively. Finally, the temperatures of water entering and leaving of the CWSC unit were almost identical to that values of CWS, CWSPP and CWSPT units.

FT-IR analysis

The infrared absorption spectrums for sand of CWS units designated as CWS, CWSPP and CWSPT in comparison with VIRGIN SAND are plotted in Fig. 5 to specify the main functional groups that enhanced the

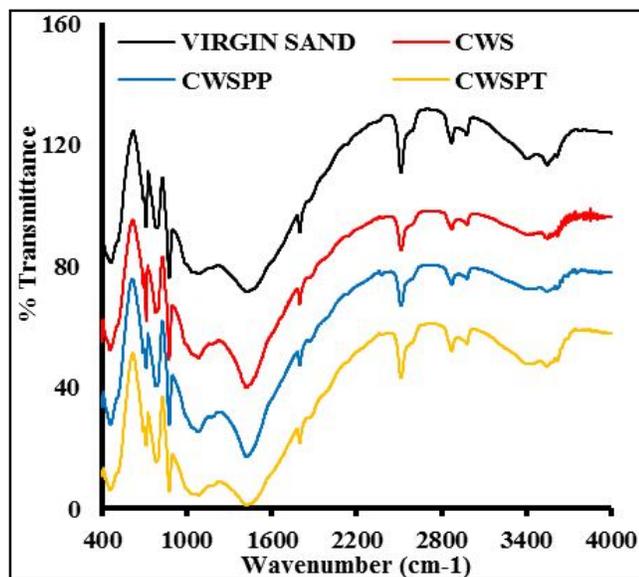


Fig. 5: Infrared absorption spectrums for sand of unplanted and planted CWs units before and after sorption of Congo red dye.

removal of dye. The stretching vibrations of hydroxyl groups (OH) can be caused high intensity absorption band at 3545.16 and 3406.29 cm^{-1} (Ahmed *et al.*, 2020) while less intense band is presented at 1797.66 cm^{-1} . Symmetrical and asymmetrical stretching vibrations can generate three bands of absorption at wave numbers of 2980.02, 2872.01 and 2513.25 (Alshammari *et al.*, 2020). Calcite can be recognized in the present samples of sand due to doubly degenerate asymmetric stretching vibration at wavenumber of 1423.47 cm^{-1} and C=O stretching at 875.68 cm^{-1} (Feng *et al.*, 2012). The Si-O-Si bond is occurred at sharp absorption band of 1083.99 cm^{-1} . The existence of Si-O symmetrical bending vibration at 794.67, 781.17 and 711.73 cm^{-1} and the O-Si-O vibrations at 692.44 and 462.92 cm^{-1} confirms the presence of quartz; however, this quartz considers crystalline due to band at 692.44 cm^{-1} (Krivoshein *et al.*, 2020).

Thus, the spectrum of FT-IR absorption just signifies an absorption bands of quartz (SiO_2) and calcite (CaCO_3) compounds in the sand samples. Therefore, it can be inferred that the dye molecules will be mainly adsorbed by SiO_2 . In addition, the changes in the intensity may be due to the interaction with functional groups of organic matter (residual of the root in sample) such as C-C, C=C and NH. The FT-IR analysis before and after sorption of Congo red illustrates the additional peaks at 1423.5 and 2872 cm^{-1} that related with C=C in the aromatic ring of this dye. Finally, the shift in other peaks indicates the adsorption of Congo red dye on the silanol group.

Conclusions

The outputs of this work certified the ability of planted and unplanted vertical subsurface flow constructed wetland unit in the reclamation of the simulated textile wastewater in terms of DO, COD and Congo red dye concentrations. For Congo red dye concentrations ranged from 10 to 40 mg/L, the removal efficiency of this dye was greater than 89% for detention time equal to 1 day and this value was increased with increase of detention time and decrease of dye concentration. Observations revealed that the plants used in the experimental units (*i.e.* *Phragmites australis* and *Typha domingensis*) have approximately the same effect on the monitoring parameters adopted to evaluate the treated wastewater quality. Also, there is slight difference in the performance of planted and unplanted units on the quality of the effluents from CWS. Clear reduction in the values of COD have been recognized to be not less than 47.4% for units under consideration at dye concentration of 10 mg/L for lowest value of contact time; however, this reduction is accompanied with a depletion in the values

of DO which consumed in the degradation process. According to guidelines of several agencies, the effluents based on the values of pH, COD, and dye concentration are suitable for irrigation purposes. FT-IR analysis proved that there are a number of functional groups enhanced the sorption of dye especially identical to SiO₂.

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