RECLAMATION OF SIMULATED CADMIUM IONS-WASTEWATER USING SUBSURFACE FLOW CONSTRUCTED WETLANDS PLANTED WITH CANNA INDICA OR TYPHA DOMINGENSIS

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ABSTRACT

This study was aimed to possibility of applying three constructed wetlands units (based on the vertical subsurface flow mode-operation) for treating of simulated wastewater contained cadmium ions. Iraqi sand was the substrate used for packing of mentioned units; however, *Canna indica* and *Typha domingensis* can be planted in any two units to improve of cadmium removal while the last one remains free of vegetation. The pH, DO, temperature, and Cd values were monitored in the effluents for detention time (0.5-120 h) and metal concentration (5-40 mgl^{-1}) to estimate the performance of units. Results proved that the removal of cadmium ions can increase significantly for longer contact time and lower Cd concentration with percentage exceeding 82% beyond 5 days. Outputs of FT-IR analysis signified the presence of several functional groups that able to improve the removal process. The values of pH, DO, temperature, TDS, and EC for treated wastewater are satisfied the requirements of irrigation water.

Keywords: heavy metal, sand material, dissolved oxygenm acidity, clean water and sensation

استصلاح مياه الصرف الصحي المحاكاة لأيونات الكادميوم باستعمال الأراضي الرطبة المُنشأة ذات التدفق الجوفي المزروعة باستعمال موز الزينة أو قصب البردي

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المستخلص

تشكل أيونات الكادميوم الموجودة في مياه الصرف الصحي الناتجة عن القطاعات الصناعية تهديدًا كبيرًا للصحة العامة والنظام البيئي بشكل عام بسبب السمية الكبيرة لهذه الأيونات. تهدف هذه الدراسة الى على امكانية تطبيق ثلاث وحدات من الأراضي الرطبة (بناءً على نمط التدفق الرأسي تحت السطح) لمعالجة المياه العادمة المحاكاة المحتوية على أيونات الكادميوم. كان الرمل العراقي هو الركيزة المستخدمة لتعبئة الوحدات المذكورة. ومع ذلك، يمكن زراعة موز الزينة وقصب البردي في أي من الوحدتين لتحسين إزالة الكادميوم بينما تبقى الوحدة الأخيرة خالية من الغطاء النباتي. تم رصد قيم الأس الهيدروجيني، والأوكسجين المذاب، ودرجة الحرارة، والكادميوم في النفايات السائلة لوقت الاحتجاز (0.5–120) ساعة وتركيز المعادن (5–40) ملغم لتر$^{-1}$ لتقدير أداء الوحدات. أثبتت النتائج أن إزالة أيونات الكادميوم يمكن أن تزيد بشكل كبير لوقت التلامس الأطول وتركيز الكادميوم المنخفض بنسبة تزيد عن 82٪ بعد 5 أيام. تدل مخرجات تحليل FT-IR على وجود عدة مجموعات وظيفية قادرة على تحسين عملية الإزالة. تستوفي قيم الأس الهيدروجيني، والأكسجين المذاب، ودرجة الحرارة، والمواد الصلبة الذائبة، والتوصيلية الكهربائية لمياه الصرف الصحي المعالجة مما يرضي متطلبات مياه الري.

الكلمات المفتاحية: عنصر ثقيل؛ مادة الرمل؛ الاوكسجين المذاب؛ الحامضية، مياه نظيفة

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INTRODUCTION

Constructed wetlands (CWs) are artificial wastewater treatment systems consisted of shallow ponds or channels. These ponds have been planted with aquatic plants. The treatment of wastewater depended upon the natural microbial, biological, physical and chemical processes (8, 21). Industrial waste water causes a lot of damage to the environments and negatively affects peoples' health. Heavy metal could be noticed from other toxic pollutant, because these metals are non-biodegradable and accumulates in the living tissue, so they concentrate throughout the foods chains (1, 2, 9), industries used cadmium, copper, lead, zinc etc. are the utmost hazardous because they generate huge quantities of metal-contaminated wastewater that disposed to the ambient. Living organisms have high ability to absorb heavy metals especially these elements can entirely dissolve in the aquatic environment. Immediately after entrance of metals to the chain of food, significant levels of mentioned metals can be accumulated in the body of human (21). Serious health disorders may be generated from ingestion the high concentration of metals that exceeding the permissible limit. So, the treatment of wastewater contained such metals prior its disposal to the environment is necessary issue. Cadmium (Cd) is the target contaminant for this study because of its toxicity that influence adversely on the health of ecosystem. Along with time, this element can be absorbed, retained and accumulated in the human body. Cd is toxic to the kidney and may also lead to the bone demineralization, either by direct bone damage or indirectly due to the renal dysfunction. In the industry, excessive exposure to airborne cadmium may increase the risk of lung cancer as a result to the impair lung function. These effects have been recorded in the communities with high exposure to cadmium in the heavily contaminated or in the industrial environment (6). The different aqueous plants like *Phragmites australis, Typhonium flagelliform, Eichhornia crapssipes, Typha, Azolla carolinian, and Lemnaetc* must be vegetated in the beds of CWs to enhance the removal of heavy metals and other toxins from wastewater. *Canna indica* (known as *phytoremediation* plant) is extensively used in the wetland establishment through several countries like China and others because it has i) large biomass, ii) rapid growth rate, iii) beautiful flowers, and iv) long root life span. This work is studied the performance of “vertical subsurface flow constructed wetlands, VSSF CWs” packed with Iraq sand to remediate the wastewater contained of cadmium element. Removal process for CW units was evaluated based on the presence of *Canna indica* and *Typha domingensis* plants in comparison with ones free from such plants through finding the reduction in the effluent cadmium concentration, as well as the variation in the dissolved oxygen (DO), temperature and pH for treated water.

MATERIALS AND METHODS

Cadmium (with atomic weight 112.41 gmole^{-1} and atomic number 48) belongs to group XII of the periodic table of chemical elements. The chemical and physical properties of this soft-silvery-white metal are similar to mercury and zinc. Synthetic polluted water samples containing maximum concentration of cadmium (40 mgl^{-1}) were prepared by dissolving $Cd(NO_3)_2.4H_2O$ in Tap water. The required mass of this compound was calculated as follows:

$$\boldsymbol{W = V \times C_i \times \frac{M{\cdot}wt}{At{\cdot}wt}} \quad \textbf{.... (1)}$$

Where W is the weight of $Cd(NO_3)_2.4H_2O$ (g), V is volume of solution (8L), C_i is initial Cd concentration, M.wt is molecular weight of $Cd(NO_3)_2.4H_2O$ (308.4 gmole^{-1}), and At.wt is atomic weight of cadmium.

The water with cadmium was prepared with different concentrations (5, 10, 20, 30, 40), and; then, this water must be injected to the planted and unplanted units to evaluate of such units in the removal of the target contaminant. This requires to withdrawn periodic samples at various time intervals like 0.5, 1, 2, 3, 4, 5, 6, 12, 24, 48, 72, 96, and 120 hours. VSSF CWs units (Fig. 1) were simulated by utilization of three containers made of plastic material with identical dimensions. Each container has bottom and top diameters of 40 and 50 cm respectively with total height of 60 cm. The container is packed with three layers of materials commonly available and suitable for CW. From the bottom of this container, coarse layer of gravel with size >10 mm and depth 15 cm were inserted to avoid the clogging of outlet; then, additional layer of fine gravel (size <10 mm) with same depth of coarse gravel must be situated on the previous layer to support the filter substrate. This substrate is represented by Iraqi sand (S) with depth of 15 cm which to be the location of plants used to improve the removal of contaminant. Each container was supplied with outlet valve fixed at height 5 cm from the bottom. This valve is used to collect the treated water samples and; also, to empty of tank. To maintain the level of wastewater within CW bed beneath the surface of substrate (obtaining subsurface system), PVC pipe of 12.5 mm in diameter was connected with outlet valve. Perforated PVC pipe (75 cm in length and 50 mm in diameter) was inserted in the CW unit through packed beds to achieve adequate aeration for substrate (3). *Canna indica* was vegetated in the first unit while *Typha domingensis* must be inserted in the second unit. This arrangement was adopted to decrease the variability in measurements and to increase practicality of the findings.

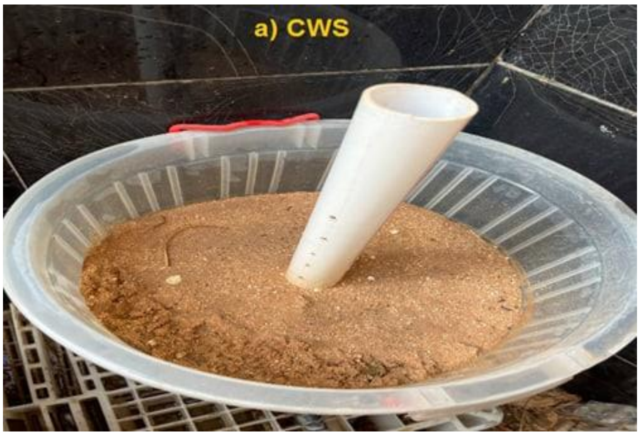


Fig. 1. Constructed wetland units (unplanted (a), planted with *Canna indica* (b), and planted with *Typha domingensis* (c)) manufactured in the present study

Vegetation: The presence of plant (either *Canna indica* or *Typha domingensis*) in VF CWs has an important role in the supporting of sedimentation and avoidance of erosion by reducing the water velocity due to increase of hydraulic pathway (13). Also, the presence of plant will cause a clear reduction in available contaminants. The growth cycle of present plants conventionally completes between March and October. When temperatures began to drop through the winter, several parts of the mentioned plants became yellow; so, must be trimmed and returned to the wetland unit. Initially, it is recommended to vegetate the plants with height ranged from 10 to 15 cm to ensure the healthy growth. Water level was reduced to enhance the deep penetration of the roots to reach the gravel layers. *Canna indica* and *Typha domingensis* are obtainable in enormous quantities through the Iraqi environment; especially, *Typha domingensis* grow in the natural wetlands located at the southern regions of Iraq. The selected plants must be submerged in the container filled with tap water for 2 days. To obtain a rapid growth, plants with 0.4 m height are always trimmed to 0.15 m; thereafter, the plants vegetated in the CW units with five plants per each unit as elucidated in Fig. 1. This process was done during November 2021.

Units operation: Wetland units operate in batch mode to reduce the expenditures like costs of pumping and automatic control. The polluted water was distributed on the top surface of the sand bed and the wastewater-sand bed contact must continue to period named “contact time”. The effluent is discharged from CW unit by "outlet valve"; then, this unit must remain in the empty status for certain period (called "resting time") between any two successive tests. The whole cycle of operation for present system is 5 days. This requires to distribute of wastewater on the bed surface through 1st day of operation after closing the "outlet valve" and maintaining the wastewater for 5 days. The samples are collected from each unit through opening the mentioned valve and taking 10 ml from aqueous solution. The outlet valve must be opened after finishing the operation to ensure that the CW free of water; finally, the system resting must extend for 10 days to get unsaturated substrate. The drainage of system from water will be accompanied with intrusion the air to the bed and re-aeration of microbes. This plan of operation can be applied for planted and unplanted vertical subsurface flow of CW units.

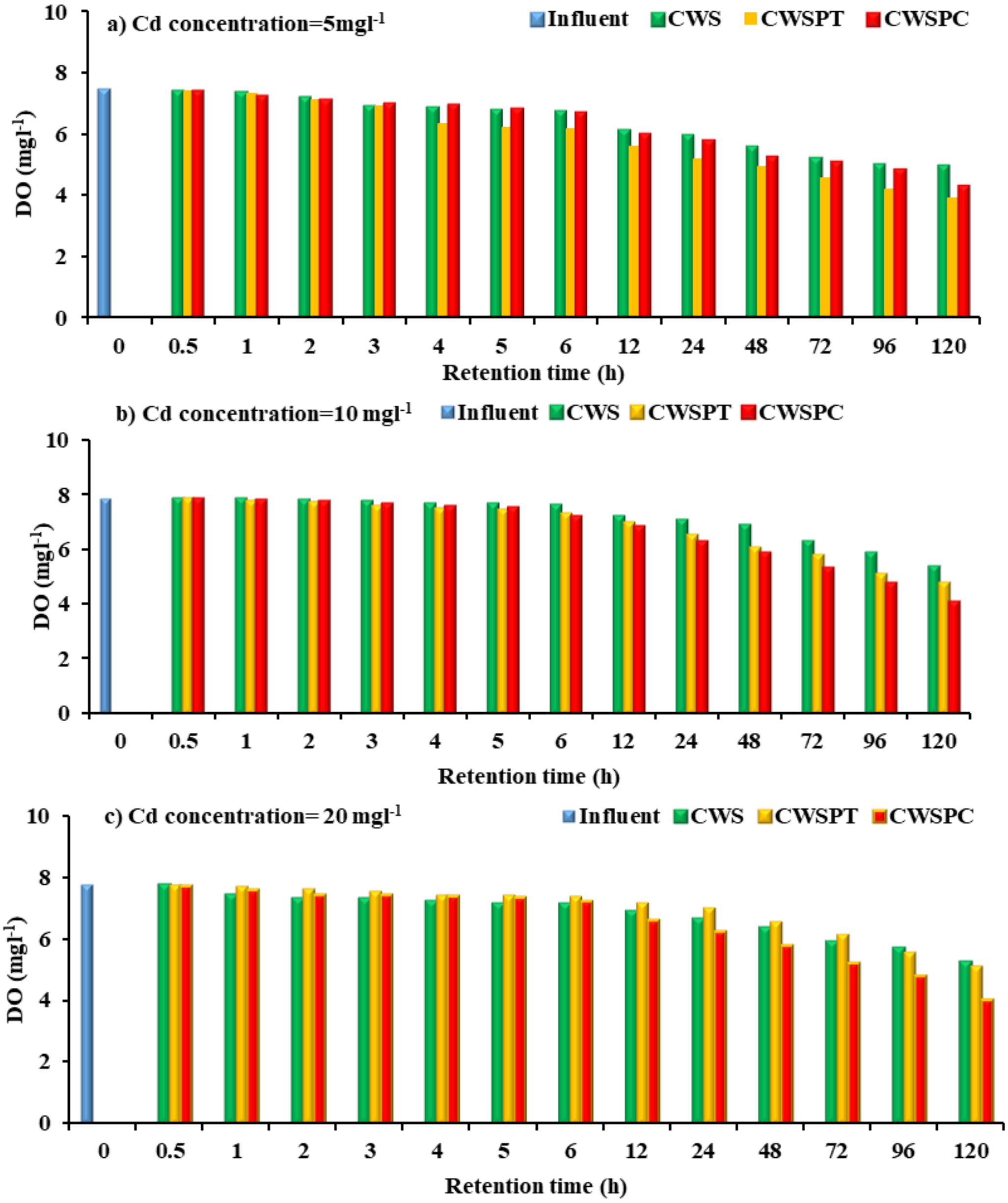
Measurement of parameters

The measurement process requires to determine cadmium concentration, dissolved oxygen (DO), pH, temperature, TDS and EC. Atomic absorption spectroscopy was applied to measure the cadmium concentration after passing the water samples through filter papers type 0.45 μm. "Hand-held mi 605 portable Dissolved Oxygen, (MARTINI, Italy)" was utilized to measure the temperature (^{o}C) and DO (mgL^{-1}). The measurements of DO were conducted immediately after sampling process to decrease the contact time with atmospheric ambient. The pH of water was determined by "hand-held E-1 portable pH electrode Digital Meter". To ensure the accuracy of pH measurement, the calibration of pH meter was achieved with a standard buffer solution of pH 4.0 and 7.0 every week. Due to the influence of pH on the chemical's solubility, the monitoring of pH is achieved. Also, TDS (mgl^{-1}) and EC (μScm^{-1}) are to measure by "hand-held E-1 portable Total Dissolved Solid and Electrical Conductivity Meter". The problems related with water quality can be reflected by values of EC where an increment in this indictor means the existence of source for dissolved ions within CW unit. Thermometer was utilized to measure the daily temperature of the ambient at the work site.

RESULTS AND DISCUSSION

Dissolved oxygen, temperature and acidity

Dissolved oxygen (DO) represents the most important indictors for removing of contaminants in the units of CWs. For present cadmium concentrations, (Fig. 2) explains that the DO of wastewater changed from 7.45 to 7.9 mgl^{-1} which identical to the initial Cd concentration varied from 5 to 40 mgl^{-1} respectively. Although this variation may not be related to cadmium concentration, it may be related to the quality of water utilized to prepare the simulated wastewater. The DO values are reduced with an increase in the retention period, due to the utilization of this oxygen in the degradation of wastewater contaminants. It appears that the vegetation kind in the CWSPC and CWSPT units (vegetated with *Canna indica* and *Typha domingensis* respectively) has not significant effect on the amount of DO in the water. Fig. 2 shows that the oxygen values in the planted CWs unit vary from 3.89 to 5.1 mgl^{-1}, but their values in the unplanted CWs unit located between 4.89 and 5.4 mgl^{-1}. The DO in the vegetated units was less over time in comparison with reference one due to higher amounts of cadmium that can oxidize and the fact that rhizobacteria consume a lot of DO (17). It is very important to explain how the pH influenced on the cadmium removal. Monitoring pH levels is crucial because of their delicate influences on the water quality, particularly dissolved ions in CWs; also, the pH may have significant influence on the microbial capacity during the breakdown of contaminants (23). World Health Organization documented that the pH is a key indicator of water quality and should be between (6.5-8.5). All cadmium concentrations used in this study (5, 10, 20, 30, and 40 mgl^{-1}) had influent pH values of 8, 7.8, 8.1, 8, and 7.8 respectively. Measurements indicated that the pH values of the effluent were slightly lower than the pH of the influent for CWS, CWSPC, and CWSPT units; particularly, during the last times of the treatment process. The influence of plants on the pH of effluent was not significant with changes not greater than 0.7. Also, records signified that the pH values in the CWSPC and CWSPT were less than that values of CWS units, this reduction may be due to phytoremediation; hence, the pH change reflects the efficient treatment of wastewater. Frequently, heavy metals are taken the ionic state for acidity conditions and these metals can precipitate under higher values of pH; so, speciation of heavy metals have greatly influenced by pH in the wetland units. The plants are removed carbon dioxide from wastewater for using it in photosynthesis and released O_2 during sunlight hours; accordingly, an increase in the pH can occur due to destabilize of carbonate ± bicarbonate equilibrium (15). All final values of pH for mentioned CWs units after 5 days indicate that they satisfy the Iraqi standards for treated wastewater. The growth of plants and microbes may continue within an appropriate temperature range. Low temperatures cause aquatic plants to wilt and go dormant, which inhibits respiration and metabolic processes including photosynthesis, lowers the synthesis and transport of OM, and prevents normal plant growth. Slight change in the effluent temperature can recognize in all CWs units and these temperatures are ranged from 21.3 to 25.7^{o}C during 5 days, while their values in the influent water varied from 21 to 23^{o}C under different concentrations of cadmium. The measured temperatures in each unit are higher than the values for the influent water's temperature and this related inversely with DO values. The increase in temperature may be correlated with an increase of microbial metabolic activity, increase carbon resource synthesis by plants, and accelerate biochemical reactions (10).



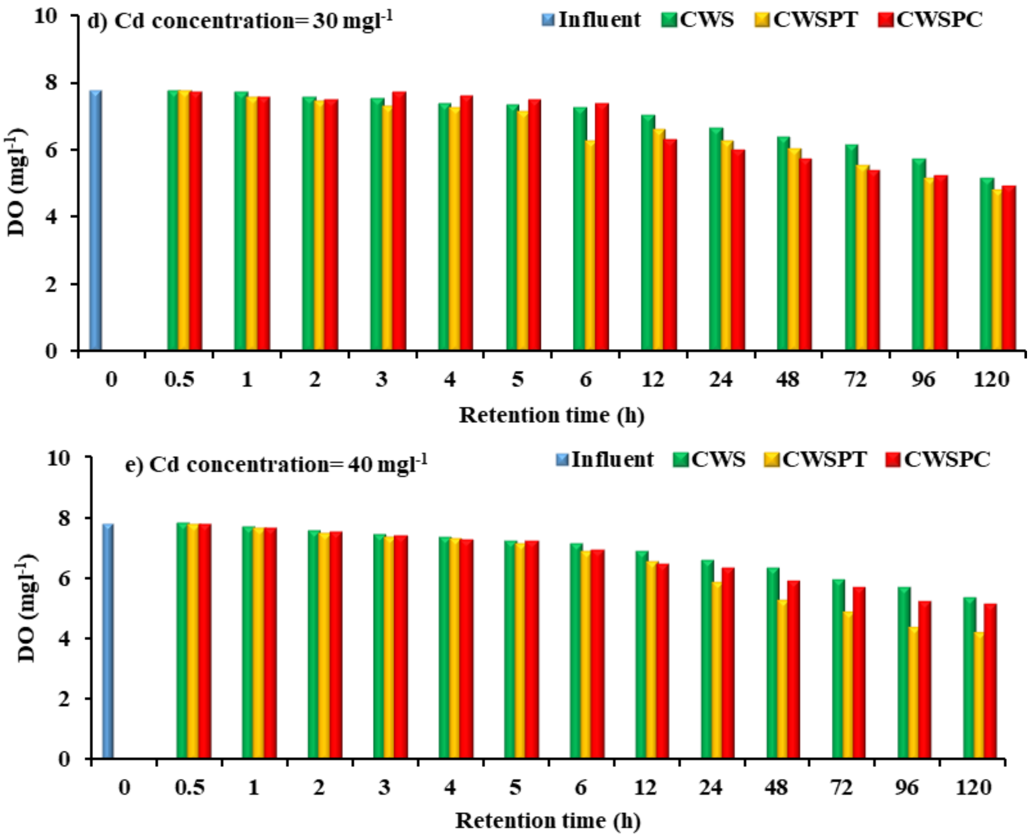


Fig. 2. The Influent and effluent values of dissolved oxygen (DO) at different influent Cd concentration (a) 5, (b) 10, (c) 20, (d) 30, and (e) 40 mgl^{-1}, through retention time of 5 days

Variation of total dissolved solids and electrical conductivity: Total dissolved solids, TDS" refer to the trace amounts of organic substances and inorganic salts that are existed in the water solution. The major components are typically carbonate, hydrogen carbonate, chloride, sulfate, and nitrate anions and calcium, magnesium, sodium, potassium, and chloride cations. The influent TDS values are with the range from 532 to 876(mgl^{-1}), and Fig. 3 showed that the TDS of effluent produced from present CWs units have values higher than that of influent wastewater. This increase may result from release of nutrients back into the water due to plant decay, increasing the content of dissolved ions and; consequently, the conductivity (16). As well, the interactions between the biofilm and substrate are the most important cause of this rise in TDS values because they release soluble salts. Additionally, the biofilms on the packed bed's particles are quickly growing and may contribute to an increase in TDS levels when they fall off and melt (20). Electrical conductivity (EC) is utilized as an indicator of water cleanliness in many industries (11). It is the most important factor in the salinity risk of a crop because it reflects the dissolved solids in wastewater. Normally, the existence of inorganic dissolved solids will affect the conductivity of water. For all of the chosen cadmium concentrations, an increase was recorded in the observed values of EC for effluents of CWS, CWSPC, and CWSPT units (maximum value 1532 μScm^{-1}) in contrast with the influent wastewater's EC values (1018 – 1088 μScm^{-1}).

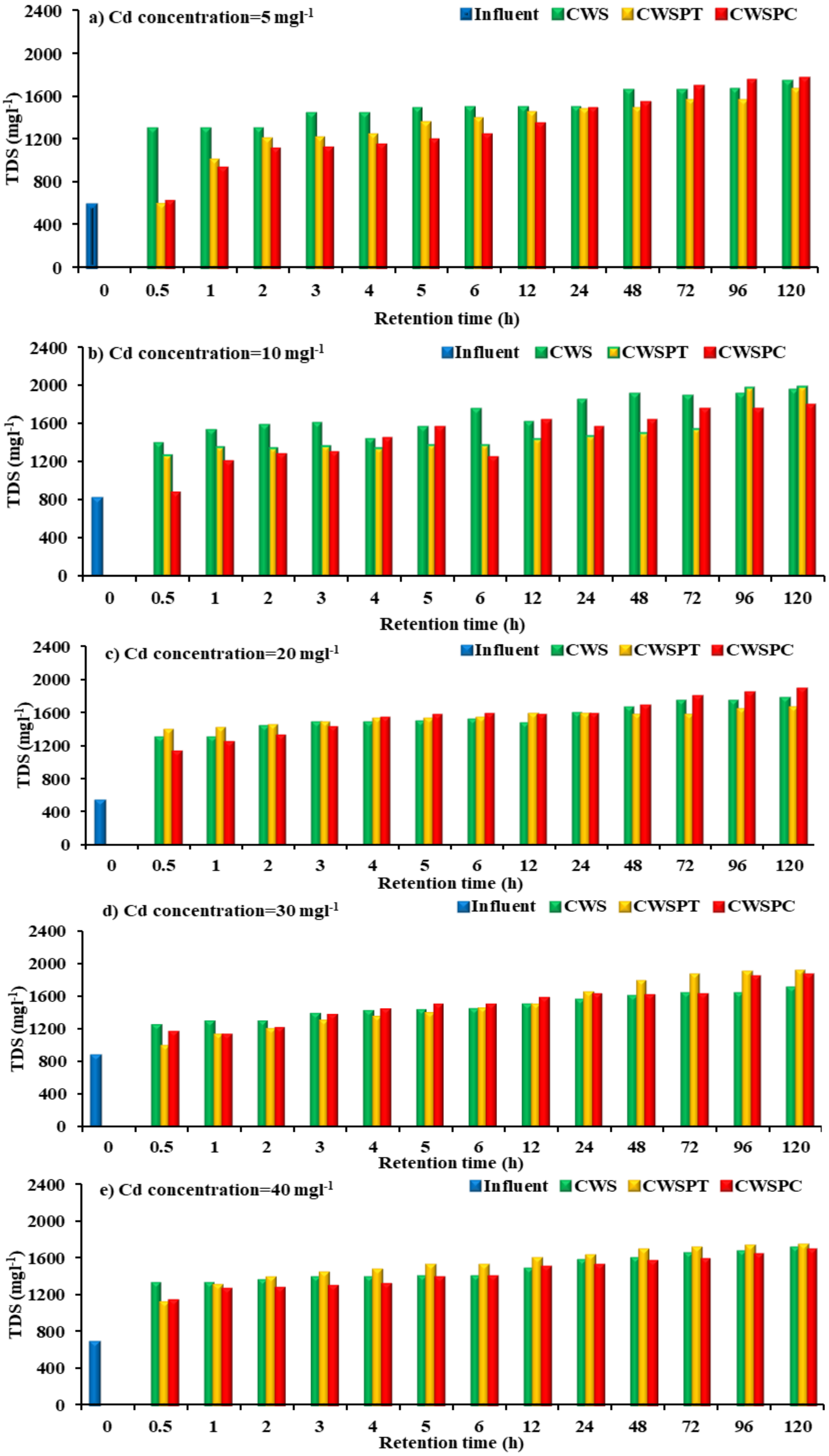
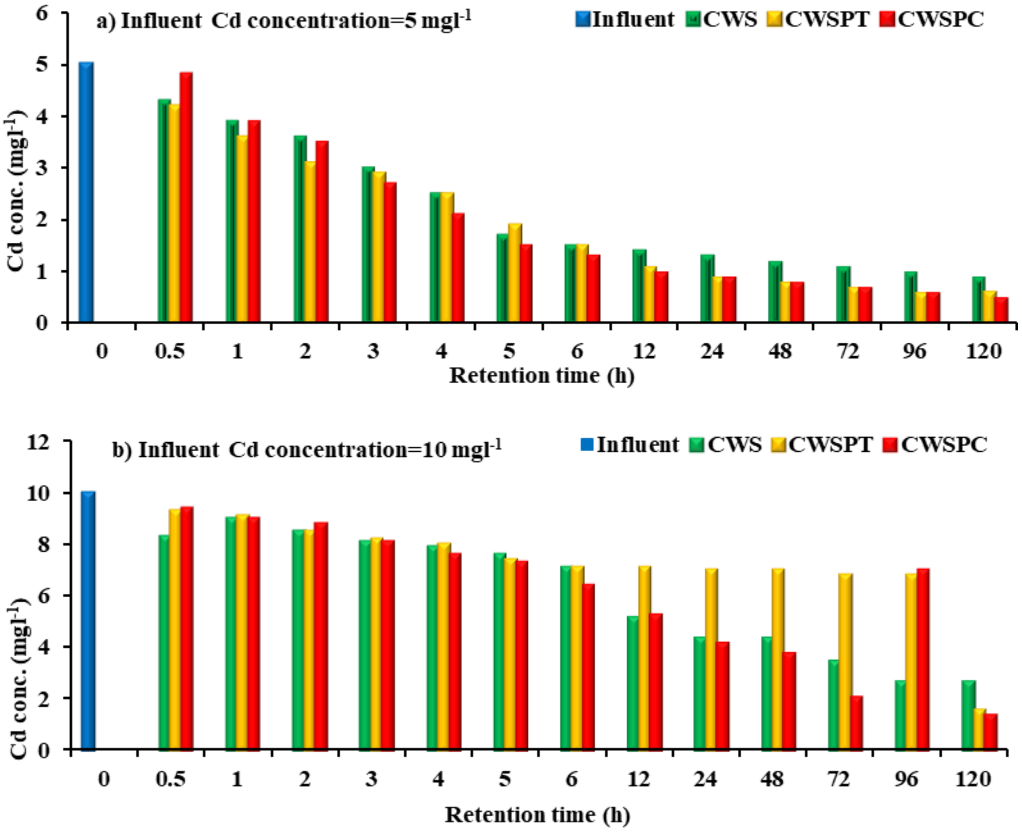


Fig. 3. The Influent and effluent values of total dissolved solids (TDS) at different influent Cd concentration (a) 5, (b) 10, (c) 20, (d) 30, and (e) 40 mgl^{-1}, through retention time of 5 days

Removal of cadmium

CWs systems are a popular and efficient way to treat wastewater that has been polluted by non-point source pollution through main five process; "sedimentation, filtration, adsorption, precipitation, and plant absorption". The removal of metal ions in conventional CWs units depends on the configuration of CWs components. The adsorption ability of substrate and the uptake characteristics of microbes in plants have an influential role in the elimination of metals as certified by previous works,where minerals accumulate in different plant parts (18). It is removed due to the bioaccumulation of minerals in the cytomembrane and cell wall (24). The vegetation of CW can remove the heavy metals from simulated wastewater either in direct pathways via phytovolatilization and phytoaccumulation, or indirect pathways through the regulation of other (4). Phytostabilization and rhizofiltration can reduce the metals mobility where the DO transfer represents the key in the sorption and microbial metabolism of metals (14). Secretions of rhizosphere contain siderophores, amino acids, organic acids, and proteins; these secretions can operate as "chelating agents" for converting of metal ions into non-toxic end-product (22). Furthermore, the mentioned secretions can reduce of redox potential with enhancement of anaerobic processes and microbial activity to regulate the metals removal. The oxygen released from the roots of plants can facilitate the metals precipitation and accelerate the degradation of nutrient (12). The performance of the VFCWs was evaluated to remove of Cd ions by applying five concentrations (5, 10, 20, 30, and 40 mgl^{-1}) to imitate the highest concentrations present in the wastewater; however, the monitoring process was extended from contact times from 0.5 hours to 5 days. Measurements signified that prepared units have significant ability in the removal of cadmium ions as plotted in Fig. 4. This figure proves that the rate of cadmium uptake in the CWSPC, and CWSPT units are greater than unplanted unit and this may be correlated with microbiological activity present in the planted units. Also, it can be noted that the removal rate decreases with the increase of the initial Cd concentration where removal percentages reached to the 82, 87.6, and 90% for the CWS, CWSPC, and CWSPT respectively for initial Cd concentration of 5 mgl^{-1}; however, these percentages have values 48, 55, and 52% for 40 mgl^{-1} of cadmium.



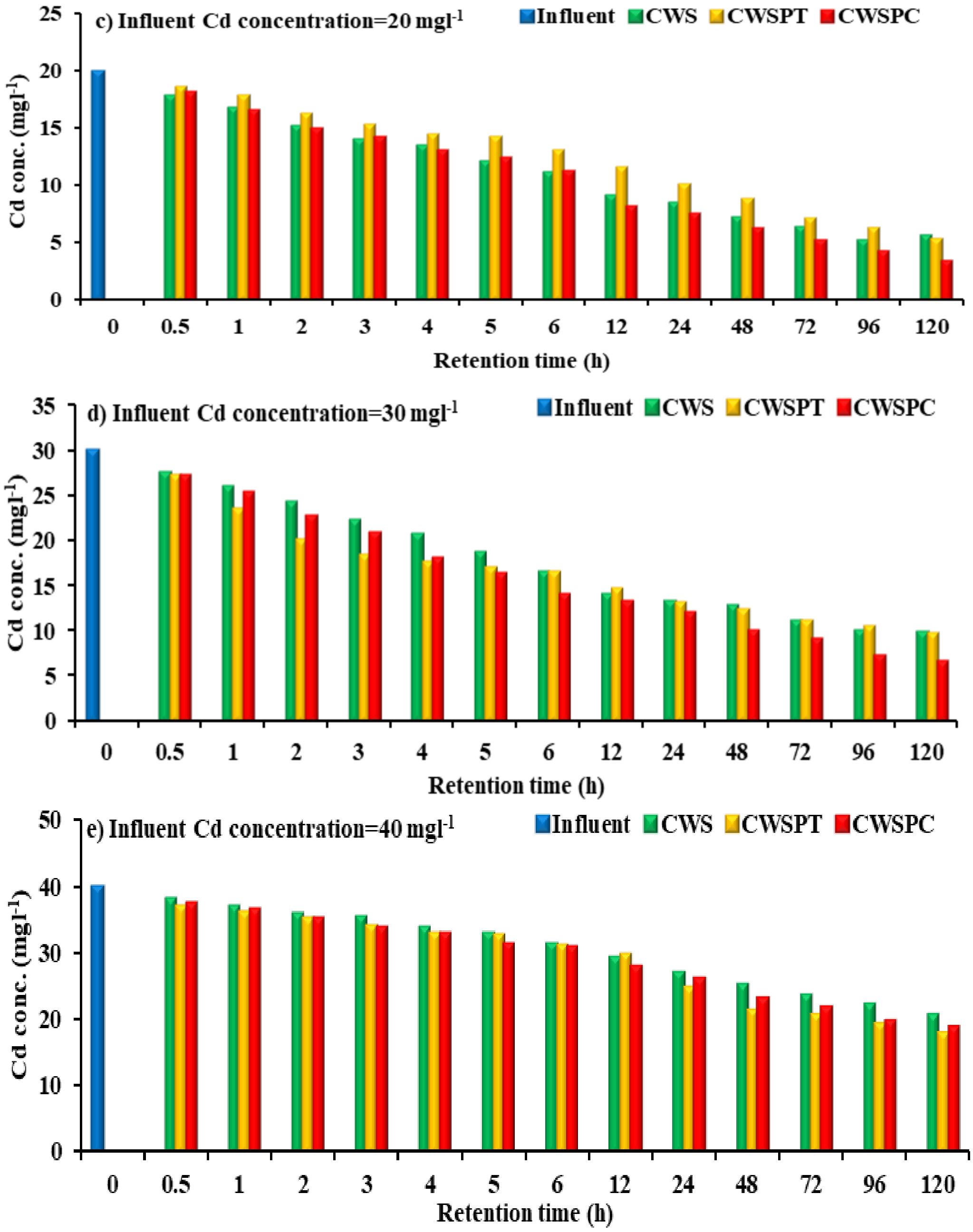


Fig.4. The Cd concentrations remaining in the wastewater treated by CWS, CWSPT, CWSPC units as function of contact time for its influent concentration of (a) 5, (b) 10, (c) 20, (d) 30, and (e) 40 mgl^{-1} during duration of 5 days

FT-IR analysis

This analysis is an analytical approach applied to specify the functional groups reflected the polymeric, organic, and inorganic substances. FT-IR technique utilizes the infrared light to scan the sample under consideration and find its chemical composition. The device of analysis depends on the sending of infrared radiation (in the region from 400 to 4000 cm^{-1}) applied on the particles of sample and the absorbed radiation can convert into vibrational energy. The signal generated in the detector represents the molecular mark of the sample, and each chemical molecule can generate a unique spectral fingerprint to identify the chemical consistent. Fig. 5 plots the FT-IR spectra for substrates of CWS, CWSPC, and CWSPT units beyond interaction with Cd ions in comparison with virgin sand to assign the prime functional groups. Fig. 5 signify that the high-intensity absorption band occurred at 3851 cm^{-1} identical to the O-H stretching vibrations (5). A less strong band, caused by the bending of H-O-H, has been seen at 1617.37 cm^{-1}. Calcite can recognize in the sand due to doubly degenerate asymmetric stretching vibration at 1461.55 cm^{-1} and C=O stretching at 875.35 cm^{-1}. The Si–O–Si bond may occur at a sharp absorption band of 1037.11, 1034.86, 1036.36, and 1033.23 cm^{-1}. The presence of O-Si-O vibrations at 669.55 and 670.1 cm^{-1} means the availability of quartz; however, this quartz considers crystalline due to the band at 472.2 cm^{-1}.A minor peak at 3611 cm^{-1} may be attributed to the presence of crystalline hydroxide. The peak at approximately 1461.55 cm^{-1} is corresponded to the CO2 vibration of calcium carbonate. The absorption band at 1650.26 cm^{-1} is represented the Si-O bending vibration (19). The peak at 3392.41, 3390.3, 3381.45, and 3374.06 cm^{-1} can correspond to the O-H strong stretching band. At wavenumber interval 2921.81 cm^{-1} stretching vibration of the C-H group is identified. Also, stretching vibrations at 3390.3 and 1037.11 cm^{-1} are described –OH in Si-OH and Si-O in Si-OH respectively where these vibrations represent the distinctive properties of silanol groups (7).

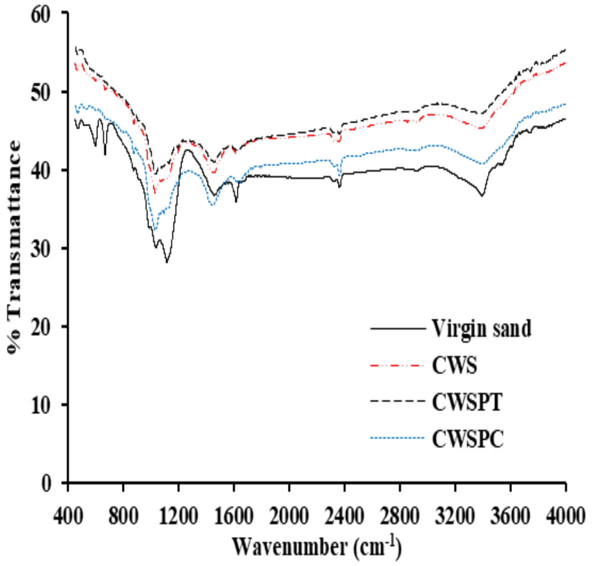


Fig. 5. Infrared absorption spectra of virgin sand in comparison with samples of substrate loaded with Cd ions treated by CWS, CWSPT and CWSPC units

Overall efficacy CWs units

The results showed that present CWs units packed with Iraqi sand have high ability in the treatment of simulated wastewater polluted with cadmium ion. The most important parameters affected on the quality of effluents are the influent concentration of Cd ions and contact time between substrate and wastewater with presence significant role for applied plant (either *Canna indica* or *Typha domingensis*). The results certified that the present units can remove high percentages of Cd ions (> 82%) for lowest concentration (=5 mgl^{-1}) and longer contact time (= 5 days); however, these percentages can decrease dramatically for same influent concentration to be less than 16% for duration of 0.5 day. Increasing of influent Cd concentration was associated with obvious reduction in the removal percentage to be < 55% for 40 mgl^{-1} influent concentration beyond 5 days. Ultimately, the quality of effluents resulted from CW units is accepted for irrigation purposes according to requirements of WHO.

CONCLUSIONS

The present study certified that the constructed wetland units with vertical subsurface flow operation mode have remarkable ability in the conversion of wastewater contained cadmium ions to water acceptable for irrigation purposes. The evaluation process was achieved through measuring of pH, DO, temperature, EC, TDS, and Cd concentration in the effluent resulted from used units for period reached to 5 days. Planted and unplanted Iraqi sand-substrate proved that more than 82% can be removed of Cd ions for lowest concentration (=5 mgl^{-1}) and longer contact time (= 5 days). The removal efficiencies have lower values (< 16%) for contact time of 0.5 day; however, an increase in the influent Cd concentration can associate with significant decrease in removal efficiency of such metal regardless the value of retention time. FT-IR for sand substrate especially planted with *Canna indica* and *Typha domingensis* proved that the reclamation of simulated wastewater is depended mainly on the available functional groups which can be enhanced greatly the removal process to satisfy the resulted water with environmental regulations.

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