Performance of Aquatic Macrophytes on Removal and Accumulation of Sulfate and Potassium from Domestic Wastewater

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Abstract Phytoremediation had been well accepted worldwide as one of the most successful green technologies for domestic wastewater treatment. This technology utilizes aquatic macrophytes to remove and extract macronutrients from the domestic wastewater and accumulate them in their plant tissues. However, most of the previous studies only focused on the use of aquatic macrophytes for removing and extracting nitrogen and phosphorous components. Thus, they overlook on sulfate (SO_4^{2-}) and potassium (K^+) as one of crucial macronutrient contaminants in domestic wastewater. Moreover, studies on removal and uptake of SO_4^{2-} and K^+ from domestic wastewater and their detail distribution and storage in macrophyte tissues are very limited. Therefore this study focused on performance of aquatic macrophytes in removing SO_4^{2-} and K^+ from domestic wastewater. The accumulation and translocation of SO_4^{2-} and K^+ throughout macrophyte bodies were also determined. The phytoremediation system was designed and fabricated in the Hydrology Laboratory in Universiti Teknologi MARA (UiTM), Shah Alam. Water Hyacinth (*Eichhornia crassipies*), Caladium (*Colocasia esculenta*) and Water Lettuce (*Pistia stratiotes*) were used in our study to demonstrate that local macrophytes may also have higher nutrients removal or uptake than other macrophytes. The performance of macrophytes in removing and accumulating SO_4^{2-}

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and K^+ and their relationship were determined and reported. The results of this study are significant for sustainable approach for domestic wastewater which would satisfy towards wholly aspect of macronutrient criteria. Hence phytoremediation process becomes alternative to compete with the other green technologies for national development.

Keywords Aquatic macrophytes · Phytoremediation system · Potassium · Sulfate · Wastewater treatment

1 Introduction

Phytoremediation had been well accepted worldwide as one of the most successful green technologies for domestic wastewater treatment. This technology utilizes aquatic macrophytes to remove and extract macronutrients from the domestic wastewater and accumulate them in their plant tissues. Thus, the phytoremediation currently becomes popular as an alternative technology in wastewater management. This is due to its ability to protect both the environment and public health in an economical way at a lower cost compared to conventional wastewater treatment [1, 2].

In recent years, there were extensxive studies on phytoremediation of macronutrients using aquatic macrophytes. These include the phyto-treatments of golf courses surface water [3, 4], anthropogenic retention ponds, lakes and wetlands [5–8], groundwater [9], swine wastewater [10, 11], dairy manure wastewater [12, 13], municipal wastewater [14] and industrial wastewater [15].

Previous studies mainly focused on the ability and efficiency of the aquatic macrophytes to remove and extract nitrogen (N) and phosphorous (P) components which were regarded as contaminants in the domestic wastewater [3, 4, 6, 11]. Although the phytoremediation system has the ability to remove N and P components effectively from wastewater, but the removal mechanisms of the other macronutrients such as SO_4^{2-} and K^+ are poorly understood. Thus, they overlook on these macronutrients as a portion of crucial contaminants in the domestic wastewater. Moreover, studies on removal and uptake of SO_4^{2-} and K^+ from domestic wastewater and their detail distribution and storage in macrophyte tissues are very limited.

In domestic wastewater, SO_4^{2-} and K^+ are regarded as abundant elements. The concentrations of SO_4^{2-} and K^+ are ranged between 20–500 mg/L [16] and 13–20 mg/L [17], respectively. SO_4^{2-} is often associated with heavy metals that affect disturbance of microbial process and nutrient imbalance in wastewater [18, 19]. Meanwhile, availability of K^+ affect the salinity and pH in wastewater [17]. These macronutrients enrichment if not well treated would cause adverse impact to the sources of water supply, biodiversity of aquatic life and contributes to water borne diseases [7, 9, 20, 21].

Therefore, this study focused on performance of aquatic macrophytes in removing SO_4^{2-} and K^+ from domestic wastewater. The accumulation and translocation of SO_4^{2-} and K^+ throughout macrophyte bodies (leaves, stems and roots) were also determined. The relationship between macrophyte species and their removal; and accumulation capacity were also determined and reported. The results of this study are significant for sustainable approach for domestic wastewater which would satisfy towards wholly aspect of macronutrient criteria. Hence phytoremediation process becomes alternative to compete with the other green technologies for national development.

2 Materials and Methods

This study was conducted in 3 stages: (a) Design and fabrication of the phytoremediation system, (b) Phytoremediation process of SO_4^{2-} and K^+, and (c) Analysis of SO_4^{2-} and K^+ in domestic wastewater and macrophyte tissues.

2.1 Design and Fabrication of the Phytoremediation System

The phytoremediation system was designed and fabricated in the Hydrology Laboratory of the Faculty of Civil Engineering in Universiti Teknologi MARA (UiTM), Shah Alam. Figure 1 shows the setup of phyto-system in the laboratory, whilst Fig. 2 is a schematic diagram of the phytoremediation system. Table 1 described details of the phyto-system as labeled in Fig. 2.

This system was designed as a batch system to integrate with the conventional wastewater treatment system when applied at site The wastewater samples were collected from Mawar Residential Sewage Treatment Plant within the Universiti Teknologi MARA (UiTM), Shah Alam. The wastewater was sampled using grab sampling method [22]. Plastic containers for sampling were thoroughly rinsed with 10 % of nitric acid and distilled water to remove contaminants. After that the wastewater was fully filled up and tightly closed in order to prevent any aeration or oxidation process [22].

Aquatic macrophytes were chosen as they were low-priced and easily available locally. They consisted of the water hyacinth (*Eichhornia crassipies*) (WH), caladium (*Colocasia esculenta*) (CD) and water lettuce (*Pistia stratiotes*) (WL). They were collected from the section 8 Lake in Shah Alam. Each of the macrophytes was cleaned thoroughly with distilled water [23], softly trashing the soft tissues, before placing them in separated containers.

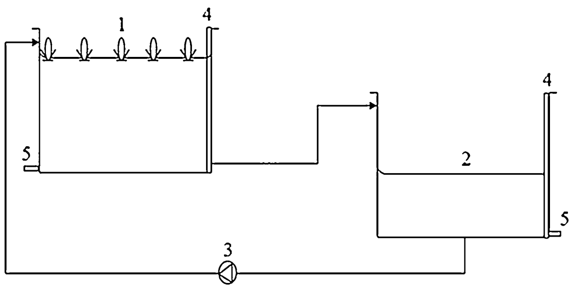

Fig. 1 Schematic diagram of the phytoremediation system

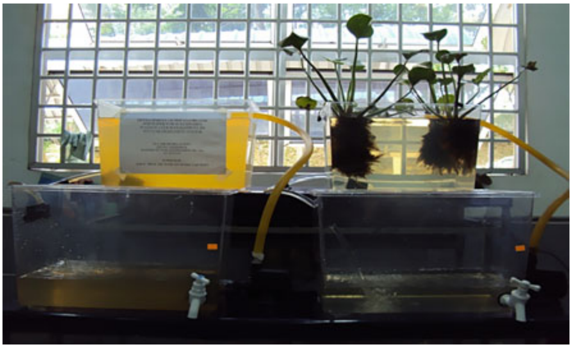

Fig. 2 Setup of phytoremediation system and control system in laboratory

2.2 Phytoremediation Process of SO_4^{2-} and K^+

The phytoremediation process was carried out immediately after the collection of wastewater samples. The wastewater samples were poured in the phyto and control systems and measured for initial level (0 day). The measurements of pH, turbidity, SO_4^{2-} and K^+ concentrations were carried out immediately within a day.

The macrophytes were introduced into the phytoremediation system. A similar system was also setup without any macrophytes to act as the control. The wastewater was initially filtered using a cotton sieve to trap suspended solids. Volume losses during the treatment due to water sampling and/or evaporation were replaced by adding distilled water [11]. The phytoremediation process was allowed to proceed until the macrophytes showed symptoms of death (survival capacity) or the nutrient concentrations in the wastewater became significantly consistent.

2.3 Analysis of SO_4^{2-} and K^+ on Domestic Wastewater and Macrophyte Tissues

The pH level of the wastewater was measured with a pH meter and the turbidity level with a turbidity meter using the procedures described by Hach Company [24]. The SO_4^{2-} and K^+ concentrations of both the phyto and control systems were

Table 1 Detail description of the phytoremediation system

No.	Apparatus/equipment	Description
1	Phytoremediation tank	Dimension: 0.66 × 0.44 × 0.35 m Material: polyvinyl chloride (PVC) Depth of wastewater: 0.22 m
2	Reservoir tank	Dimension: 0.66 × 0.44 × 0.35 m Material: polyvinyl chloride (PVC) Depth of wastewater: 0.09 m
	Total volume of wastewater (both tanks)	24 L
3	Water pump	Type: super 300 multi-use pump Head of pressure: 2.2 m Flow of wastewater: 6 L/min
4	Level meter	–
5	Discharge valve	–

determined by using a Spectrophotometer DR5000 according to the Sulfaver 4 Method (8031) and Tetraphenylborate Method (8049) [24], respectively.

As for macrophyte tissues analysis, phytoremediated aquatic macrophytes were transferred into different containers, according to their species before being cleaned with saline water (2 M NaCI), aqueous ethylenediaminetetraacetic acid (EDTA) (2 g/L), tap water and deionized water [25], softly trashing with tissues. They were then sorted into leaf, stem and root parts. Each separated macrophyte bodies of roots, stems and leaves were dried at ambient temperature (27 °C) before oven dried at 80 °C for 48 h. The dried tissues were weighed and grounded to powder form [11]. The dried tissues (1.0 g dry weight) were digested using Milestone Ethos Plus according to acid sulfuric digestion method with 8 ml of HNO_3 65 % and 2 ml of H_2O_2 30 % via published procedure [26]. The samples after digestion were cooled to room temperature. Next, they were filtered through the Whatman No. 1 filter paper and then 0.45 µm membrane filters in order to reject the suspended substances. Later, they were analyzed for SO_4^{2-} and K^+ using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) (Perkin Elmer Optima 7300 DV).

3 Data Analysis

Removal rate (%) of SO_4^{2-} and K^+ were determined using the following formula:

$$R\ (\%) = (C_0 - C_t)/C_0 \times 100\,\% \quad (1)$$

where C_0 is the initial concentration of SO_4^{2-} and K^+ (mg/l) and C_t is the concentration of SO_4^{2-} and K^+ (mg/l) at time t (days). The data were analyzed for mean, standard deviation (n = 3) and error bar with 5 % of value using the statistical package within Microsoft® Excel Version 2010.

4 Results and Discussion

Table 2 presents initial and final concentrations and removal rate of pH, turbidity, SO_4^{2-} and K^+ for phyto and control systems.

4.1 Performance of Aquatic Macrophytes in Changing pH and Turbidity Levels from Domestic Wastewater

The pH level in WH system was at alkaline range for 1-day to 4-day (highest 8.44) and change to acidic state after 5-day (lowest 5.83) until end of treatment. The pH level in CD system was at alkaline state started from 1-day to 15-day (highest 7.91) and the pH level was reduce towards acidic state for 16-day to 24-day (lowest 6.31) and remain alkaline state until end of treatment. The pH level was kept within an alkaline range (highest 7.78) in water lettuce system. The pH level in control system differs within a day, where at alkaline state from 1-day to 4-day, reduce to acidic state from 5-day to 15-day and back to alkaline state from 16-day to 30-day (Fig. 3).

The turbidity of the phyto-systems in the WH, CD and WL reduced from 43.97 NTU to 0.86 NTU, 57.50 NTU to 0.64 NTU and 58.40 NTU to 0.83 NTU, respectively compared to control systems which reduced from the level of 57.93 NTU to 1.31 NTU (Table 2). While the removal rate of the phyto- systems in the WH, CD and WL were 98.04, 98.89 and 98.58 %, respectively compared to removal rate of the control system was 97.74 % (Table 2).

The changes of pH level in phyto-systems were moved toward stability or consistency, compared to control system. This is due to the utilization of the carbonate compounds during the algal photosynthesis [6], where macrophytes enhanced this process via root system. The pH values were decrease to acidic state for both WH (at the end of treatment) and CD systems (day-16 to day-24) could be explained by the oxidation of wastewater organic matters, the nitrification of N containing compounds [27] and CO_2 is dissolved from the atmosphere and produced by decomposition of organic matter. It is readily soluble in water, combining with the water to form carbonic matters [28].

Turbidity levels in the phyto and control systems were significantly declined at beginning of treatment (1-day) where CD system was able to reduce more turbidity level higher than the other systems. The results also prove that the sole system itself able to reduce the turbidity level with evidence of the reduction in the control system. Besides, the turbidity level was more enhanced with the presence of macrophytes in the phyto-system. The reason behind this fact is that the roots of the macrophytes were proficient to trap particulate matters during the phyto-treatment [11]. It also has been reported that abundant root system in the phyto-treatment could secretes large amount of root exudates. They are polymeric in nature and assists in the coagulation and sedimentation processes of the suspended particles by destabilizing the colloidal suspensions [29].

Table 2 Initial and final concentrations and removal rate of pH, turbidity, SO_4^{2-} and K

Parameters		Phytoremediation systems			Control systems
		WH	CD	WL	
Initial concentration	pH	6.99 ± 0.00	7.31 ± 0.08	7.24 ± 0.00	7.31 ± 0.08
	Turbidity	43.97 ± 0.15	57.50 ± 3.27	58.40 ± 4.11	57.93 ± 3.29
	SO_4^{2-}	15.67 ± 2.08	15.67 ± 0.58	8.00 ± 0.00	14.00 ± 3.61
	K^+	8.23 ± 0.06	6.03 ± 0.06	6.23 ± 0.06	6.03 ± 0.06
Final concentration	pH	5.83 ± 0.17	7.05 ± 0.06	7.77 ± 0.04	7.57 ± 0.05
	Turbidity	0.86 ± 0.57	0.64 ± 0.08	0.83 ± 0.05	1.31 ± 0.04
	SO_4^{2-}	12.67 ± 0.58	6.00 ± 0.00	5.00 ± 0.00	13.00 ± 1.00
	K^+	37.10 ± 0.35	0.60 ± 0.02	14.45 ± 0.00	16.50 ± 0.04
Removal rate (%)	Turbidity	98.04	98.89	98.58	97.74
	SO_4^{2-}	19.15	61.71	37.50	7.14
	K^+	−77.82	90.05	−56.89	−63.46

Mean ± standard deviation (n = 3)

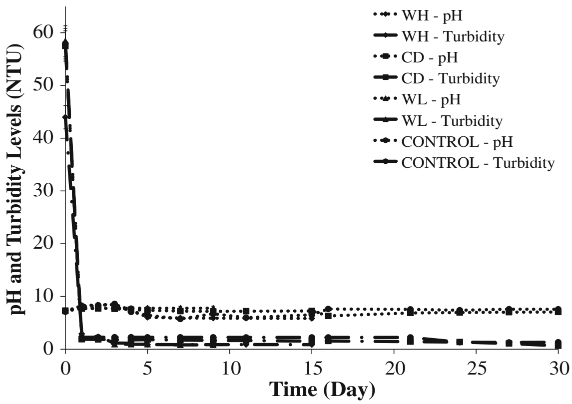

Fig. 3 Changes of pH and turbidity levels during phytoremediation process

4.2 Performance of Aquatic Macrophytes in Removing SO_4^{2-} and K from Domestic Wastewater

After the period of treatment, WH, CD and WL effectively remove 61.71, 37.50 and 19.15 % of SO_4^{2-} in the phyto-systems, respectively, whilst control system removes 7.14 % at the end of treatment period (Table 2). Besides, WH and WL in the phyto-systems and control system showed increments of 77.82, 56.89 and 63.46 % of K^+, respectively. Somehow, in CD system, the level of K reduced to 90.05 % at the end of phyto-treatment (Table 2).

Although macrophytes in the phyto-systems had a limited effect on SO_4^{2-}, but they could perform as stabilizer for transformation of SO_4^{2-} in wastewater process. This was verified by the trend of SO_4^{2-} in phyto-systems were more stable than the control system, which values of SO_4^{2-} in the control system was fluctuate

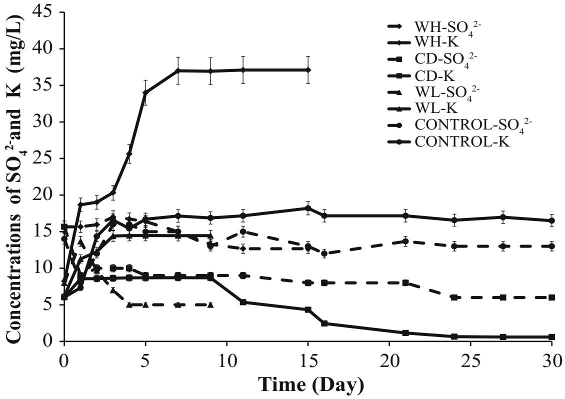

Fig. 4 Changes of SO_4^{2-} and K^+ concentrations during phytoremediation process

within a day (Fig. 4). The fate of S in the wastewater is mainly governed by the redox state dynamic in the root system, with the availability of organic carbon and O_2 [30, 31] and sulfate-reducing bacteria [32].

Under aerobic condition in the phyto-systems and control system, the loses of SO_4^{2-} caused by oxidation of sulfide (S^{2-}) to SO_4^{2-} and dissociation reactions of S^{2-} to hydrogen sulfide (H_2S) which is largely volatile and emitted to atmosphere [33]. Increase of pH (to alkaline state) in the initial stage of the experiments (Fig. 3) indicated that H_2S emitted to the atmosphere [34]. Loses of SO_4^{2-} was also due to heavy metal precipitation to form insoluble metal sulphates (such as $FeSO_4$, $ZnSO_4$) and mineral precipitation to form mineral sulphates (such as $CaSO_4$), when SO_4^{2-} was more favorable than S^{2-} in wastewater [33]. Furthermore, with the presence of the macrophytes could enhance the sulphates precipitation process.

In addition, phyto-systems demonstrated higher reduction of SO_4^{2-} than control system since macrophytes provide a suitable roots zone for biological assimilation into macrophyte tissues or microorganism biomass as organic S [33]. They also released O_2 via root zone for direct re-oxidation of S^{2-} to SO_4^{2-} [32] and stimulate microorganism growth and activity [35]. The uptake of SO_4^{2-} also is more favored compare to S^{2-} since it is highly toxic to macrophytes and microorganisms. S^{2-} is also competitor for O_2 consumption [33, 36], hence it mainly present in anaerobic condition.

Nevertheless, there are no consistencies of K^+ concentration in the both phyto and control systems. However, CD system shows positive trends of K^+ removal (Fig. 4). It have been stated that the K^+ concentration rose as natural wetland communities tended to export K^+ [3, 37]. Somehow, they did not mention the factors of K^+ rises. Thus, in the present study, K^+ level rising most probably resulted from degradation of macrophytes tissues during phyto-treatment process. This was in line when the macrophytes were reached at aging phase (mostly degraded).

The availability of K^+ in the wastewater also was affected by salinity and pH in wastewater [17]. This is because of the K^+ in the wastewater is related to hardness by proportion of $CaCO_3$. However, the present results revealed that there were no significant relationship between pH and K^+ since pH levels for both phyto and control systems moved towards stability.

4.3 Accumulation and Translocation of S and K Throughout Macrophyte Bodies

At the end of the experiment, CD exhibited higher accumulation capacity for S and K of 157.03 and 1,937.69 mg/L, respectively, compared to S and K in WH of 200.93 and 1,530.95 mg/L; and WL of 75.76 and 83.67 mg/L, respectively (Fig. 5).

Although Wu et al. [32] reported that S is essential for macrophytes growth which supposed to be equal or larger than P, somehow in this study the contents of K in the all of examined macrophytes tissues were higher than the S (Fig. 5). This heading towards that the K was not limiting growth of the macrophytes compared to S.

As S^{2-} may lead to phytotoxic effects, macrophytes in the phyto-system more favor SO_4^{2-}. Hence the uptake of S was mainly by SO_4^{2-}. Thus, S was detected in the macrophyte tissues in this study leading to verify the above statement. Somehow, some of macrophytes show deterioration effect, such as WH and WL, as they may uptake of metal sulphates (such as $FeSO_4$, $ZnSO_4$). These metal sulphates especially $FeSO_4$ have toxic effects on the macrophytes since Fe^{2+} is also phytoxin [33]. As a result, it reflected that macrophytes capable to prevent phytoxin by accumulated most of S in below-ground mass before distribute to above-ground mass (Fig. 5).

It has been reported that K^+ was important macronutrients for macrophyte growth, especially for biomass yield and foliar area [14], thus K^+ was translocated higher in above-ground mass compared to below-ground mass (Fig. 5). This case was contradict with the statement that macrophytes has a problems in uptake base cations such as K^+ when they are normally occur in rich anion environment (e.g. NO_3^-) as their main macronutrient source [38]. Besides, K^+ has excellent water solubility thereby it is easy to mobile throughout macrophytes tissues but very difficult to precipitate.

4.4 Relationship Between Macrophytes Species and Their Performance Against Removal and Accumulation of S and K

Relationship between removal and accumulation rates for S and K^+ of WH and WL were inversely proportional. Both of WH and WL performed well in removing SO_4^{2-} and accumulating K^+. But then, negative rate were noticed for removal of

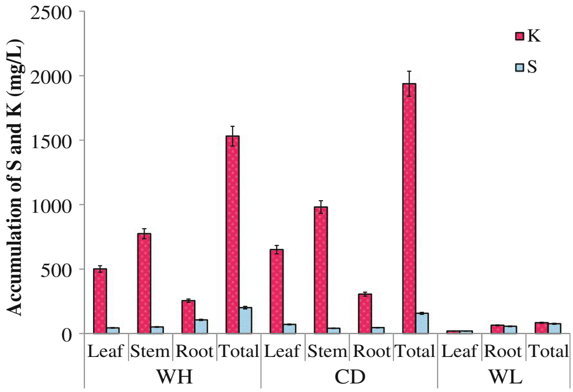

Fig. 5 Distribution of S and K throughout macrophytes bodies

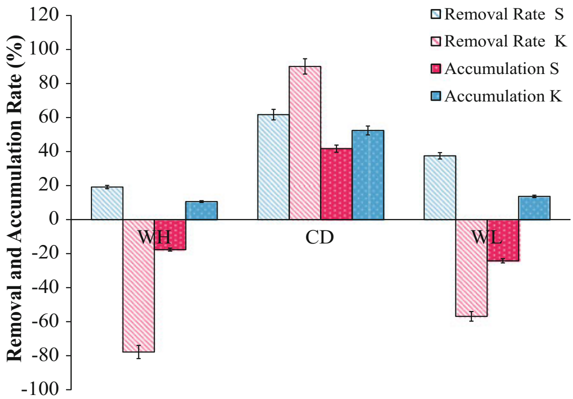

Fig. 6 Performances of macrophytes based on removal and accumulation rate of S and K

K^+ and accumulation of S. Conversely, CD performed better in removing and accumulating of both S and K^+ (Fig. 6). Thus, make CD was the best removal and accumulator species of S and K^+ rather than others species. Performance of macrophytes on SO_4^{2-} and K^+ removal was also in relation with their physical and biomass characteristics.

It has been stated that macrophytes with higher potential capacity for biomass production could perform better macronutrients removal [39]. It appears that S was difficult to mobile throughout macrophyte bodies compared with K^+. On the other hand, SO_4^{2-} was constantly removed in domestic wastewater by phyto-systems.

5 Conclusions

In conclusion, the performances of the three aquatic macrophytes in removing and accumulating the SO_4^{2-} and K^+ from domestic wastewater using phytoremediation system were evaluated. CD displayed more capacity to reduce and accumulate SO_4^{2-} and K^+ compared to the other macrophytes. Thus, the results have indicated that the selection of aquatic macrophytes is a crucial stage in determining the performance in phytoremediation system. Subsequently, this proves that different macrophyte species have variation abilities for macronutrients removal and uptake.

The authors also recommend that the relationship between macrophyte survival and growth in the phytoremediation system need to be established. This is an important measurement criterion since the growth of plants influence the removal and uptake mechanisms of the plants.

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