Abstract. Water pollution by heavy metals has currently been an increasing worldwide concern. This study was conducted to assess the removal efficiency of a treatment system using sorbent and common reed (Phragmites australis). Approximately 1400 g of sorbent that was modified from Ban Cuon iron mine drainage sludge by adding 10% sodium silicate solution and heating at 400 °C was put in a column of 1.8l. The initial concentrations of As and Cd in inflow water were 1.0 mg/l, those of Pb, Mn, and Zn were 6.0 mg/l. During 30 days of the laboratory experiment, aqueous solution of constant flow rate (50 l/day) was pumped through the sorbent column before flowing into surface and horizontal subsurface constructed wetlands using P. australis. The results showed that the removal efficiency of As, Cd, Mn, Pb and Zn by sorbent – surface constructed wetland were 83.0 – 89.5, 89.3 – 93.3, 69.7 – 98.0, 94.8 – 95.4, and 69.2 – 92.3%, respectively; those by sorbent – subsurface constructed wetland were 85.3 – 89.1, 98.5 – 98.7, 92.8 – 98.7, 95.5 – 96.0, and 93.1 – 96.2%. The concentrations of heavy metals in the outlet water met the National Technical Regulation on Industrial Wastewater of Vietnam (QCVN 40:2011–BTNMT) with the exception of As, which indicates the effectiveness of combined sorbent and P. australis for simultaneous removal of these metals from contaminated water.

Keywords: constructed wetland; heavy metals; iron mine drainage sludge; Phragmites australis; removal.

1. Introduction

Water quality is a major challenge in the 21st century. Approximately 90% of all wastewater in developing countries is discharged into the surrounding environments without proper treatment [1]. As a consequence, pollutants accumulated in aquatic ecosystems in surface water, groundwater, soil, and plants [2][3][4]. Heavy metals are becoming one of the most serious environmental problems [5].

Wastewater treatment by constructed wetlands (CWs) is a biotechnological process that has been proved to be efficient in removing some chemicals (e.g., organic substances, and heavy metals) and biological organisms (e.g., bacteria, viruses, and parasites) from municipal and domestic wastewaters [6][7]. Due to low energy requirements, convenient operation, and simple maintenance, CWs may enable the cost–effective and ecological treatment of agricultural, industrial, and municipal runoff wastewaters [8][9][10]. A wide range of aquatic plant species (e.g., Lemna minor L., Eichhornia crassipes, Polygonum amphibium L.) has been reported to accumulate heavy metals [11]. Among them, common reed (P. australis) is one of the most widely distributed species in the world, and has
been frequently used in CWs for treatment of industrial wastewaters containing metals in the last two decades [12]. However, CWs may require a larger land area than a conventional facility [13][14][15]. Therefore, the preliminary treatment by other treatment technologies may be needed for enhancing removal of heavy metals from contaminated water. Adsorption has emerged as a cost–effective and efficiency alternative pre–treatment for the removal of heavy metals if low–cost and environment–friendly materials are used [5][16].

A combined sorbent and CWs using P. australis has recently been used for treatment of wastewater contaminated with medium concentrations of heavy metals [17]. This study aimed to assess the efficiency of this combined sorbent and CW system for removal of heavy metals at higher concentrations in inflow water. The results of this study could assist to design the treatment system practices with a range of metal concentrations in the inlet water.

2. Materials and Methods

2.1. Plant, sorbent, and inlet water preparation

The present study was used the same sorbent and CWs as those reported by Ha and Anh [17]. The biomass of P. australis was designed to increase approximately 30%. Reed blocks were also more developed and distinctly branched. For inlet water preparation, a mixture solution of heavy metals was prepared by mixing nitrate salts of Mn, Zn, Cd, Pb, and As with de–ionized water to obtain the desire concentrations. The initial concentrations of As and Cd in inflow water were 1.0 mg/l, those of Mn, Zn, and Pb were 6.0 mg/l. The pH of solution was adjusted to pH 7 by using HNO₃ and NaOH.

2.2. Experimental setup and sampling

During 30 days of the laboratory experiment, aqueous solution of constant flow rate (100 l/day) was pumped through the sorbent column before separated evenly into surface and subsurface CWs (Figure 1). All the design parameters of the experimental system were set the same as those reported by Ha and Anh [17] (Table 1).

| Characteristics                  | Values              |
|----------------------------------|---------------------|
| Total sorbent column height      | 230 mm              |
| Column diameter                  | 100 mm              |
| Total sorbent weight             | 1400 g              |
| Hydraulic retention time         | 30 min              |
| Substrate                        | Natural soil        |
| Volume                           | 0.175 m³            |
| Surface area                     | 0.35 m²             |
| Porosity of substrate            | 55%                 |
| Safety factor                    | 2%                  |
| Hydraulic retention time         | 48 h                |
| Substrate                        | Gravel (2–4 cm grain size) |
| Volume                           | 0.21 m³             |
| Surface area                     | 0.42 m²             |
| Porosity of substrate            | 45%                 |
| Safety factor                    | 2%                  |
| Hydraulic retention time         | 48 h                |

Input and output water samples of sorbent column, surface and subsurface CW systems were collected before and at 1, 3, 5, 7, 10, 13, 16, 19, 22, 25, and 30 days of the experiment. Sorbent and
plant samples were taken at the beginning and the end of the experiment. In total, 44 water samples, 9 plant samples, and 3 sorbent samples were collected.

![Figure 1. Experimental design of sorbent column and constructed wetland](image)

2.3. Analytical methods

*P. australis* was well rinsed with deionized water. Samples of plant, and sorbent were dried in a ventilated oven at 80 °C for 48 hours. The dried samples of plants were ground into fine powder using a mortar mill. The dried sorbent were crushed into fine powder using the MRC laboratory Equipment Manufac User. Sorbent and plant samples were digested with mixture (HF:HNO$_3$:HCl = 1:1:2) and (H$_2$O$_2$:HNO$_3$ =1:5), respectively. Microwave digestion of sorbent and plant samples was performed by Multiwave pro Anton Paar. Concentrations of heavy metals in sorbent, plant, and water samples were determined by Atomic Absorption Spectrometer (AAS, Agilent 240FS with hydride generation accessory VGA77) at the VNU University of Science, Vietnam National University, Hanoi.

2.4. Equations

The amount of heavy metals sorbed by sorbent ($C_s$) was determined by the following equation:

$$C_s (mg) = (C (mg/kg) - C_o (mg/kg) \times m_s (kg)) \times 100 (l/day)$$

Where $C_o$ is the concentration of heavy metal in sorbent before the experiment, $C$ is the concentration of heavy metal in sorbent after the experiment, $m_s$ is the amount of sorbent used in the experiment.

The amount of heavy metals that removed by sorbent column ($C_r$) after 30 days of experiment was determined by the following equation:

$$C_r (mg) = (C_l (mg/l) - C_r (mg/l) \times 30 (days) \times 100 (l/day))$$

Where $C_l$ is the concentration of heavy metal in water before entering the sorbent column, $C_r$ is the concentration of heavy metals in water discharging from sorbent column.

Total amount of heavy metal accumulated in *P. australis* ($C_a$) after 30 days of experiment was determined by the following equation:

$$C_a (mg) = (C_o (mg/kg) - C_o (mg/kg) \times m_b (kg/m^2) \times s (m^2))$$

Where $C_o$ is the concentration of heavy metal in the plant before the experiment, $C'$ is the concentration of heavy metal in plant after the experiment, $m_b$ is the estimated biomass of *P. australis* used in the experiment, $s$ is the surface areas of CWs.

Total amount of metal removed by CWs ($C'_r$) was determined by the following equation:

$$C'_r (mg) = (C'_l (mg/l) - C'_r (mg/l) \times 30 (days) \times 100 (l/day))$$

Where $C'_l$ is the concentration of heavy metal in water before entering the CW systems, $C'_r$ is the concentration of heavy metals in water discharging from CWs.

The bioconcentration factor (BCF) is defined as the ratio of the total concentration of the element in the whole plant to that in the growing solution [18]. The translocation factor (TF) is defined as the ratio of element concentrations in the stems to that in the roots [19].
3. Results and Discussion

3.1. Removal of heavy metals by sorbent and constructed wetlands

The removal efficiency of As, Cd, Mn, Pb, and Zn by sorbent – surface CW were varied within 83.0 – 89.5, 89.3 – 93.3, 69.7 – 98.0, 94.8 – 95.4, and 69.2 – 92.3%, respectively; those by sorbent – subsurface CW varied within 85.3 – 89.1, 98.5 – 98.7, 92.8 – 98.7, 95.5 – 96.0, and 93.1 – 96.2% (Figure 2). The results demonstrated that the removal rates of heavy metals by subsurface flow CW were higher than those by the surface flow system.

![Figure 2](image-url)

The concentrations of Mn in the inlet of sorbent column were 6.0 mg/l which decreased to 1.37 mg/l after passing through the sorbent column on the first day and 5.08 mg/l on the 30th day of the experiment (Figure 2). Removal rates of Mn by sorbent on the first day was 92.0%, which decreased to 15.3% on the last day of the experiment. Removal rates of Mn in sorbent column – surface flow CW and sorbent column – subsurface flow CW were 92.8 and 98.0% after 3 days, which decreased to 98.7 and 69.7%, respectively on the last day of the experiment.

The removal efficiency of Zn by sorbent column on the first day was 93.0%, which decreased to 41.7 after 15 days, and dropped to 12.0% at the last day of the experiment. However, the removal rates...
of CWs were relatively stable which indicated the long term remediation of Zn by sorbent – CW systems. Zn removal efficiency in the first and the last day of experiment by sorbent column – subsurface flow CW was 96.2 and 93.1%, and that by sorbent column–surface flow CW was 92.3 and 69.2%.

The concentrations of As in the outlet of sorbent column increased steadily during 30 days of experiment, ranging from 0.23 to 0.58 mg/l (Figure 2). In contrast, removal rates of As by sorbent decreased from 77.5 to 42.5%. Respectively, removal rates of As in sorbent column–surface flow CW and sorbent column–subsurface flow CW were 89.5 and 89.1% after 3 days, 87.3 and 87.2% after 15 days, 83.0 and 85.3% after 30 days of experiment. Constant concentrations of As in the outlet water indicated the stability of As removal by the systems.

The concentrations of Cd in the outlet of sorbent column increased steadily during 30 days of experiment (Figure 2). However, the combined system showed relatively high efficiency for Cd removal. The average Cd removal rates of sorbent column–surface flow CW and sorbent column–subsurface flow CW were 93.3 and 98.5% respectively. After 30 days of the experiment, removal rates of Cd by both systems remained high with 89.3% and 98.7%, respectively.

The concentrations of Pb in the inlet of sorbent column was 6.0 mg/l which decreased to 0.28 mg/l on the first day and 3.23 mg/l on the 30th day of the experiment (Figure 2). This indicated the higher removal of Pb by sorbent column than that of Mn, Cd, Zn, and As. Pb removal efficiency by sorbent column was 93.5% for the first day of the experiment which decreased to 46.1% after 30 days of the experiment. Removal efficiency of Pb in sorbent column – surface flow CW and sorbent column – subsurface flow CW after 3 days were 95.4% and 96.0%, which decreased to 94.8% and 95.5%, respectively after 30 days of the experiment.

The concentrations of Zn and Pb in the outlet water of sorbent column – surface CW system met the allowable limits by Vietnam regulation for industrial wastewater type B (QCVN 40:2011–BTNMT – B) [20] during the experiment, those of Mn and Cd after the 15th day of the experiment exceeded the regulation QCVN 40:2011–BTNMT – B [20] (Figure 2). In sorbent column–subsurface CW system, the concentrations of Mn, Zn, Cd in the outlet water were lower than the regulation QCVN 40:2011–BTNMT – A [20] during the experiment; however, the concentrations of Pb only fulfilled the regulation within the first 15 days of the experiment. The results of this study demonstrated the capacity of combined sorbent and CWs system for treatment of wastewater contaminated with heavy metals (Mn, Zn, and Pb ≤ 6 mg/l; Cd ≤ 1 mg/l). The concentrations of As in the outlet water exceeded the allowable limits regulated at QCVN 40:2011–BTNMT – A, B [20] (Figure 2). This fact required the alternatives of sorbent with higher As removal efficiency or using different substrates at CWs, such as laterite or zeolite. The experiment conducted by Allende et al. [21] using horizontal flow wetland planted with P. australis showed that the removal efficiency of As was 99.9% by zeolite wetlands. The concentrations of heavy metals in the outlet water were compared with the integrated wastewater discharge standard by Ministry of Ecology and Environment of China (GB 8978–1996) [22]. The results showed that the concentrations of Mn and Zn in the outlet water met the limits which are 2.0 and 1.0 mg/l, respectively. The concentrations of Pb, Cd, and As were higher than those regulated in the GB 8978–1996, which are 0.1, 0.01, and 0.1 mg/l, respectively.

| Table 2. Concentrations of heavy metals in water in two experiments |
|---------------------------------------------------------------|
| **Experiment** | **Concentrations of heavy metals in water (mg/l)** |
|                | Mn | Zn | As | Cd | Pb |
| Ha and Anh [17] | 4.0 | 1.5 | 0.4 | 0.1 | 0.6 |
| Inlet water    | 0.753 | 0.375 | 0.088 | 0.054 | 0.272 |
| Surface CW    | 0.610 | 0.363 | 0.083 | 0.037 | 0.216 |
| Subsurface CW | 1.818 | 1.850 | 0.174 | 0.126 | 0.393 |
| This study     | 0.126 | 0.449 | 0.149 | 0.028 | 0.280 |
| Inlet water    | 0.5 | 3 | 0.05 | 0.05 | 0.1 |
| Surface CW    | 1 | 3 | 0.10 | 0.10 | 0.5 |
| Subsurface CW | 0.5 | 3 | 0.05 | 0.05 | 0.1 |
| QCVN 40:2011–BTNMT – A [20] | 0.5 | 3 | 0.05 | 0.05 | 0.1 |
| QCVN 40:2011–BTNMT – B [20] | 1 | 3 | 0.10 | 0.10 | 0.5 |
Bold values indicate values that exceeded the regulation limits for industrial wastewater.

In the previous study, Ha and Anh [17] conducted the laboratory experiment with concentrations of Mn, Zn, Pb, and As in the inlet water were 4.0, 1.5, 0.6, and 0.4 mg/l, respectively (Table 2). In comparison, with higher concentrations of heavy metals in the inlet water, the removal efficiency of sorbent – subsurface flow CWs system also showed better performance than those in the sorbent – surface flow system. The concentrations of Mn, Zn, Pb, and As in the outlet water from sorbent – CW systems in the previous study [17] were lower than the allowable limits QCVN 40:2011–BTNMT – B [20] (Table 2). The results of this study demonstrated lower removal efficiencies of heavy metals sorbent column than those reported by Ha and Anh [17]; however, higher and more stable removal rates of heavy metals by sorbent column – CWs were obtained (Table 3). This is possibly due to an increase in biomass of P. australis in both surface and subsurface CWs in the present study which may result in higher removal of heavy metals by CWs.

Table 3. Removal efficiency of heavy metals from water in two experiments

| Heavy metal | Day of experiment | Removal rates of heavy metals (%) |
|-------------|-------------------|----------------------------------|
|             |                   | Sorbent – subsurface CW          |
|             |                   | Sorbent – surface CW             |
|             |                   | Ha and Anh[17] This study         |
|             |                   | Ha and Anh[17] This study         |
|             |                   | Ha and Anh[17] This study         |
| Mn          | 3                 | 67.6                              |
|            | 15                | 33.8                              |
|            | 30                | 30.6                              |
| Zn          | 3                 | 69.5                              |
|            | 15                | 56.3                              |
|            | 30                | 51.1                              |
| Cd          | 3                 | 67.5                              |
|            | 15                | 85.5                              |
|            | 30                | 68.7                              |
| Pb          | 3                 | 91.5                              |
|            | 15                | 49.0                              |
|            | 30                | 22.8                              |
| As          | 3                 | 42.0                              |
|            | 15                | 49.2                              |
|            | 30                | 39.8                              |

3.2. Accumulation of heavy metals in sorbent

The concentrations of Mn, Zn, Cd, Pb, and As in the sorbent before the experiment were 1460, 930, 170, 490, and 67 mg/kg which increased to 2442, 1646, 390, 1053, and 246 mg/kg, respectively after 30 days of the experiment. Using the equation (1), the total amounts of Mn, Zn, Cd, Pb, and As sorbed during 30 days of the experiment were 982, 716, 220, 563, and 179 mg, respectively (Figure 3). The total amounts of Mn, Zn, Cd, Pb, and As removed by sorbent column in 30 days calculated by equation (2) were 4582, 2734, 223, 884, and 512 mg, respectively (Figure 3). The ratios of metal amount sorbed to sorbent to that removed from solution by sorbent column were varied greatly (Mn – 18.8%, Zn – 24.3%, Cd – 78.3%, Pb – 57.8%, and As – 26.9%) (Figure 3). These ratios were higher than those reported by Chinh et al. [23], possibly due to the lower concentrations of heavy metals in the inlet water in this study. This result indicated that in addition to sorption of heavy metals to sorbent, precipitation of heavy metals in the surface material (co–precipitation) or inner–sphere complexation, outer–sphere complexation can be occurred [24][25]. This is in accordance with Thang et al. [26] that using the similar sorbent for fixed bed column sorption of heavy metals.

3.3. Accumulation of heavy metals in P. australis
The results of this study showed that the concentrations of Mn, Zn, Pb, Cd, and As in the roots of *P. australis* in subsurface CW were 2490, 434, 217, 110, and 40.5 mg/kg–DW, respectively; much higher than those in the stems which were 75.1, 103, 116, 44.2, and 3.2 mg/kg–DW. Higher concentrations of heavy metals in *P. australis* in the roots than those in the stems were also observed in surface CW. This is in agreement with previous findings of Bonanno and Giudice [27], who found the main bioaccumulation trend in plant organs according to which metal concentrations decrease in the order of roots > rhizomes > leaves > stems. The concentrations of Mn, Zn, Pb, Cd, and As in the roots of *P. australis* in surface CWs were 1060, 758, 330, 94.4, and 17.1 mg/kg–DW, respectively; those in the stems were 65.7, 153, 127, 48.0, and 3.5 mg/kg–DW (Figure 4). The concentrations of Mn, Zn, Pb, and As in the plant roots in the present study were lower than those reported to be 3920, 1020, 1350, and 183 mg/kg–DW, respectively [28].

![Figure 3](image3.png)  
**Figure 3.** Total amounts of metal removal and sorbed to sorbent

![Figure 4](image4.png)  
**Figure 4.** Accumulation of heavy metals in *P. australis* (a) (b)

![Figure 5](image5.png)  
**Figure 5.** Total amounts of metal accumulated in *P. australis* and removal by subsurface CW (a) and surface CW systems (b)

The estimated biomass of *P. australis* was 5 kg/m² in dry weight (DW). Using equations (3) and (4), the amounts of heavy metals accumulated in the plant and the total amounts of heavy metals removed from the wastewater by the surface CWs and subsurface CWs were obtained (Figure 5). In subsurface CW system, the amounts of Mn, Zn, Cd, Pb, and As accumulated in the plant were 3980, 718, 255, 379, and 54.7 g, respectively. The total amounts of Mn, Zn, Cd, Pb, and As removed from the wastewater by the CWs after 30 days of experiment were 7950, 3270, 660, 2880, and 360 g, respectively. In surface CW system, the amounts of Mn, Zn, Cd, Pb, and As accumulated in the plant were 1760, 1650, 277, 711, and 39.5 g, respectively. The total amounts of Mn, Zn, Cd, Pb, and As removed by the CWs were 5580, 4710, 540, 2640, and 330 g, respectively (Figure 5). This result indicated that several processes (e.g., adsorption to substrates, co-precipitation to root, filtration and deposition in the rhizosphere, metal uptake and accumulation in *P. australis*) can be involved in metal removal in the CWs [29][30].

The bioconcentration factor (BCF) values of Cd, Mn, Pb, Zn, and As in subsurface CW were 2610, 1970, 1110, 558, and 120, respectively; those in surface CW were 2140, 520, 987, 759, and 68 (Table
4). The BCF values in subsurface CW were in the following order: Cd > Mn > Pb > Zn > As, those in surface CW were Cd > Pb > Zn > Mn > As (Table 4). The BCF values of both surface and subsurface CWs in this study were much higher than those reported by Nga and Ha [28]. This may reflect the difference in biomass of *P. australis* and the difference in initial concentrations of Mn, Zn, Cd, Pb, and As that were 4, 1.5, 0.1, 0.6, and 0.4 mg/l, respectively [28].

**Table 4. Bioconcentration factor and translocation factor**

| Bioconcentration factor (BCF) | Mn | Zn | Cd | Pb | As |
|------------------------------|----|----|----|----|----|
| **This study**               |    |    |    |    |    |
| Surface CW                  | 520| 759| 2140| 987| 38 |
| Subsurface CW               | 1970| 558| 2610| 1110| 120|
| **Nga and Ha, 2016**        |    |    |    |    |    |
| Surface CW                  | 112| 111| 130| 253| 187|
| Subsurface CW               | 0.08| 0.17| 0.50| 0.44| 0.30|

| Translocation factor (TF)   |    |    |    |    |    |
|------------------------------|----|----|----|----|----|
| **This study**               |    |    |    |    |    |
| Surface CW                  | 0.02| 0.25| 0.72| 0.62| 0.10|
| Subsurface CW               | 0.10| 0.27| 0.30| 0.84| 0.92|
| **Nga and Ha, 2016**        |    |    |    |    |    |
| Surface CW                  | 0.10| 0.27| 0.30| 0.84| 0.92|
| Subsurface CW               | 0.10| 0.27| 0.30| 0.84| 0.92|

The translocation factor (TF) values in subsurface CWs were higher than that in surface CWs for Zn, Cd, and Pb. TF values of 5 heavy metals in this study were less than 1, indicating the poor capacity of *P. australis* to transport heavy metals from the roots to the above parts of the plants (Table 4).

4. Conclusions

The combination of modified iron mine drainage sludge and surface flow or subsurface flow CWs showed potential for removal of heavy metals from water. The removal efficiencies of heavy metals by sorbent – subsurface flow CWs system showed better performance than those by sorbent – surface flow system. Heavy metals concentration in the outlet water met the allowable limits for industrial wastewater type B (QCVN 40:2011–BTNMT – B) with the exception of As which indicated further studies on enhancing treatment of water contaminated with As.

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