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Performance comparison of gravel- and oil palm shell-based constructed wetland mesocosms for copper, zinc and lead removal

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ABSTRACT Heavy metal pollution in aquatic environment usually happens at low concentration. Current heavy metal removal technologies have high operational costs. Subsurface flow constructed wetland is a potential solution as it is a low-cost ecotechnology. The high production of underutilized oil palm shell in Malaysia poses a potential as an alternative media for constructed wetland in heavy metal removal. This study aimed to compare the feasibility of oil palm shells as alternative media to gravel in mesocosm-scale constructed wetland for the removal of copper, zinc and lead. All oil palm shell- and gravel-based mesocosm units, unplanted and planted with *Typha angustifolia*, were spiked with synthetic wastewater under batch drain-and-fill mode and operated at three days hydraulic retention time for 180 days. The removal of lead in both oil palm shell- and gravel-based mesocosms were consistently near 100%. Copper and zinc removals in the oil palm shell-based mesocosms were ranging at 93.11 - 100% and 97.75 - 100%, respectively. Whereas copper removal in the gravel-based mesocosms ranged at 92.13 - 99.98%. Although pH in the planted gravel-based mesocosm was near neutral (pH 6.47 - 7.43), plant presence reduced zinc removal from 99.93% (Day 228) to 66.37% (Day 405) in the gravel-based mesocosm. Higher removal of sulphate in the oil palm shell-based mesocosms suggested the occurrence of metal sulphideprecipitation. Oil palm shell-based mesocosms performed better than gravel-based mesocosms, both in unplanted and planted conditions. Further studies need to be done on the changes of media's physicochemical properties after experiment as well as copper, zinc and lead mass balance to understand better the removal pathway of copper, zinc and lead in all the mesocosms.

KEYWORDS: Oil palm shell; Constructed wetland; Heavy metal; *Typha angustifolia*; Wastewater treatment
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Original Article

INTRODUCTION

Heavy metal pollution is omnipresent in our surrounding environment yet often went unnoticed as heavy metal pollution usually occurred at low concentration (Ashraf *et al*., 2020; Ihunwo *et al*., 2020). However, the non-degradable and bio-accumulative behaviour of heavy metal in food chain raised concerns on its potential adverse health effect (Afonne and Ifediba, 2020; Chen *et al*., 2018). The commonly used commercial wastewater treatment technology such as membrane filtration (Huang *et al*., 2017) and chemical precipitation (Hu *et al*., 2017) were deemed costly to treat low concentration of heavy metal.

Constructed wetland - an eco-technology - is a prospective alternative wastewater treatment system to treat heavy metal polluted wastewater (Bakhshoodeh *et al*., 2017; Engida *et al*., 2020; Jia *et al*., 2020; Mustapha *et al*., 2018). Moreover, constructed wetland can treat low concentration heavy metal at low cost (Liu *et al*., 2020). Constructed wetland mimicked the physical, chemical and biological processes as well as the basic components' (media, water body, plant and microbe) interactions of the natural wetland under controlled condition (USEPA, 2000; Vymazal, 2018; Vymazal, 2005).

The constructed wetland is generally categorized into subsurface (SSF) and free water surface flow (FWS) (Chong *et al*., 2024; Wu *et al*., 2015). The SSF constructed wetland was preferred for heavy metal removal compared to the FWS constructed wetland as heavy metal removal is higher in reducing condition (Pedescoll *et al*., 2015) and it does not prone to mosquitoes breeding issue (USEPA, 2000). The SSF constructed wetland is referred to as constructed wetland hereafter. Studies have reported that copper (Cu), zinc (Zn) and lead (Pb) removal pathways in constructed wetland were precipitation, co-precipitation and adsorption (Machemer and Wildeman, 1992; Marchand *et al*., 2010; Zhang *et al*., 2020). Heavy metal was often retained in the constructed wetland's basin (Dan *et al*., 2017; Papaevangelou *et al*., 2017). Gravel has been conventionally used as constructed wetland media which filled up the basin and there was little information on utilization of alternative media such as recalcitrant organic waste which motivated this work.

Malaysia was the world's second largest palm oil producer (FAO, 2024) with Sabah state being the largest producer in Malaysia as of July 2024 (MPOB, 2024). The oil palm industry generated large volume of waste, one of which is oil palm shell, leading to waste management issues. Chong *et al*. (2013) and Jong & Tang (2015) reported the application of oil palm shell as constructed wetland media and its ability to remove heavy metal. This suggested its potential as a cheaper alternative, constructed wetland media for the treatment of heavy metal, concurrently solving its disposal problem. The objective of this study was to compare the performance of gravel- and oil palm shell-based constructed wetland mesocosms, unplanted and planted with *Typha angustifolia*, in Cu, Zn and Pb removal.

METHODOLOGY

Preparation of Media

Pea size gravel was purchased, cleaned with tap water, soaked overnight with distilled water to remove impurities. Spent oil palm shell was collected from a local oil palm mill. It was then extensively washed, rinsed and soaked in batches to remove impurities. The gravel and oil palm shell were separately air-sun dried and kept in clean plastic gunny respectively. The cleaned gravel and oil palm shell were respectively referred to as G and OPS hereafter.

Construction of Constructed Wetland Mesocosm Units

Polypropylene tanks with the dimension of 62.7 x 40.3 x 40.4 cm (length x width x height) were assembled with a support structure made of 12 mm waterproof plywood. The position of inlet, sampling port, outlet and *Typha angustifolia* rhizomes in the constructed wetland units is shown in Figure 1. The constructed wetland media was filled to 30 cm height to be in line with the water level. Healthy *Typha angustifolia* rhizomes were collected from a nearby drainage system and immediately transported to study site for cleaning, documentation and transplantation in the constructed wetland mesocosms (Day 0). The *Typha angustifolia* planted constructed wetland mesocosms which media were OPS and G, respectively, was codenamed as OPSP and GP. Meanwhile, the unplanted constructed wetland mesocosms which media were OPS and G, respectively, were codenamed as OPSU and GU. The OPSU, OPSP, GU and GP were placed outdoor exposed to actual tropical condition. All mesocosm units were applied 5 g/week of fertilizer (N:P:K 15:15:15), operated under five days hydraulic retention time (HRT) and allowed to acclimatize and mature for 224 days.

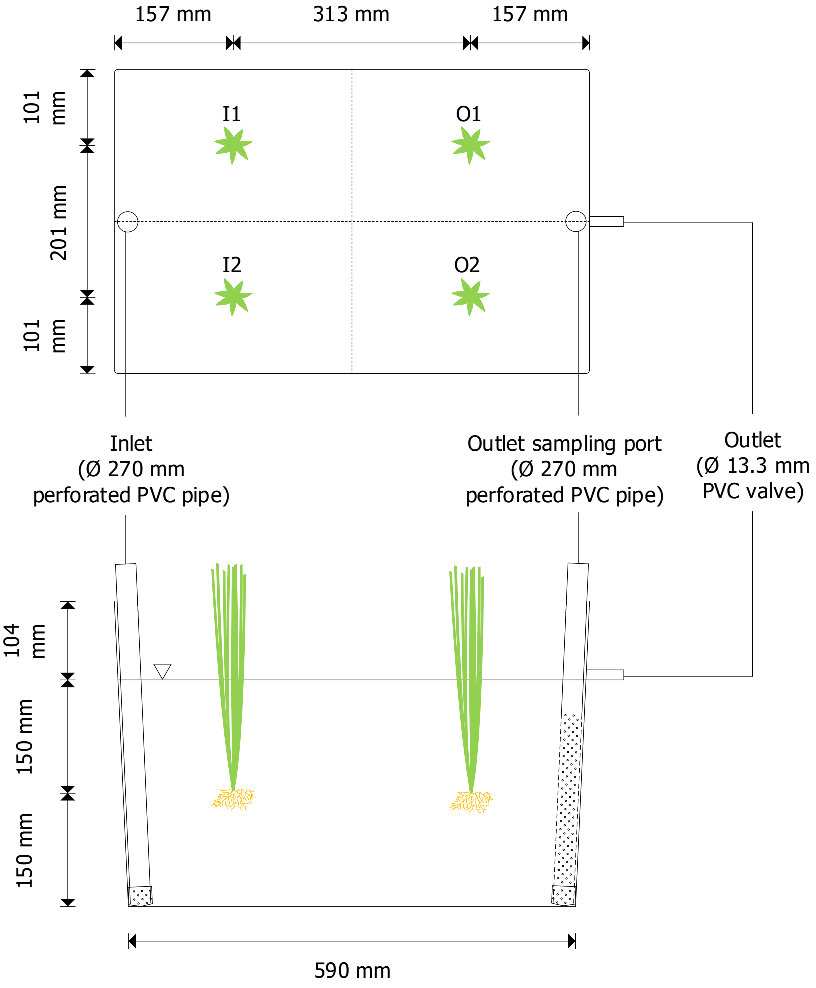

Figure 1. Top and cross-sectional views of constructed wetland unit

Treatment of Copper, Zinc and Lead

Upon maturation, all mesocosm units were spiked with synthetic wastewater containing 5 mg/L Cu, 10 mg/L Zn and 2.5 mg/L Pb (Lim *et al*., 2003) and experimented outdoor for 180 days (Day 225 – 405). All mesocosm units were operated in batch drain-and-fill mode under the HRT of three days (60 cycles). Treated wastewater samples were taken from the outlet sampling port of each constructed wetland unit at the end of each operating cycle (Figure 2). pH, temperature and sulphate (SO_4^{2-}) content of wastewater samples were determined immediately. The samples were then filtered (Membrane Solutions, mixed cellulose ester (MCE) membrane filter, 0.45 μm, Ø 47 mm) to remove turbidity, preserved with 65% (w/w) HNO_3 to pH < 2 and analyzed with five points (0.1, 0.3, 0.5, 0.7 and 1.0 mg/L) calibrated induced coupled plasma optical emission spectrometer (Perkin Elmer Optima 5300 DV) using diluted samples from the 21 Elements Quality Control Standard (Perkin Elmer, 100 mg/L, 5% (w/w) HNO_3).

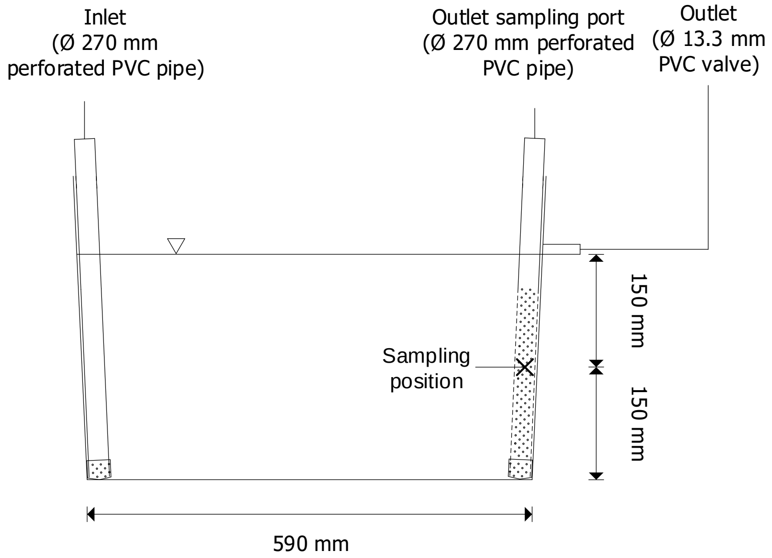

Figure 2. Sampling position of treated wastewater

RESULTS AND DISCUSSION

The pH, Temperature and SO_4^{2-} Level in OPSU, OPSP, GU and GP

The mean pH of effluent from OPSU, OPSP, GU and GP were 6.47 ± 0.18, 7.09 ± 0.17, 6.90 ± 0.41 and 7.02 ± 0.21, respectively (Table 1). The presence of plants in OPSP and GP resulted higher pH due to the production of root exudates (Jiang *et al*., 2023). Plant root uptake of carbon dioxide (CO_2) in the mesocosm and thus the pH rose (Faisal *et al*., 2023; Siriwardhana *et al*., 2023). The absence of plants in the OPSU and GU resulted in higher CO_2 content in the waterbody which led to the production of H^+ as described in Eqs. 1 – 3 (Thomas *et al*., 2022).

Table 1. The pH, temperature and SO_4^{2-} level in the OPSU, OPSP, GU and GP

Parameter	**Constructed wetland mesocosm**							
	Influent				Effluent			
	OPSU	OPSP	GU	GP	OPSU	OPSP	GU	GP
pH	3.96 (0.11)	3.97 (0.11)	3.98 (0.14)	3.97 (0.14)	6.47 (0.18)	7.09 (0.17)	6.90 (0.41)	7.02 (0.21)
Temperature (°C)	28.1 (1.8)	28.1 (1.8)	28.1 (1.9)	28.1 (1.8)	27.8 (2.1)	27.8 (2.1)	27.9 (2.0)	27.8 (2.1)
SO_4^{2-} (mg/L)	32 (7)	32 (7)	32 (8)	32 (7)	1 (4)	0 (1)	22 (8)	19 (9)

Value in parentheses denotes standard deviation

$$CO_2\ (aq) + H_2O\ (aq) \leftrightarrow H_2CO_3\ \ (aq) \tag{1}$$

$$H_2CO_3\ \ (aq) \leftrightarrow H^+\ (aq) + HCO_3^-\ (aq) \tag{2}$$

$$HCO_3^-\ (aq) \leftrightarrow H^+\ (aq) + CO_3^{2-}\ (aq) \tag{3}$$

The average temperature of OPSU, OPSP, GU and GP from Day 225 – 405 were 27.8 ± 2.1, 27.8 ± 2.1, 27.9 ± 2.0 and 27.8 ± 2.1 °C, respectively. This was typical of the tropical weather. The mean SO_4^{2-} value of effluent in OSPU and OPSP were 1 ± 4 and 0 ± 1 mg/L, respectively. This indicated possible occurrence of H_2S production by sulphate reducing bacteria in OPSU and OPSP, which could cause metal sulphide precipitation. The presence of organic matter (carbon) sourced from the OPSU and OPSP media acted as electron donor and allowed sulphate reduction by sulphate reducing bacteria at pH ranging between pH 5.34 – 6.46 (Chen *et al*., 2021a) as shown at Eqs. 4 and 5 (Machemer and Wildeman, 1992; Wu *et al*., 2013).

$$SO_4^{2-}\ (aq) + 2CH_2O\ (aq) \rightarrow H_2S\ (g) + 2HCO_3^-\ (aq) \quad (4)$$

$$H_2S\ (g) + M^{2+}\ (aq) \rightarrow MS\ (s) + 2H^+\ (aq) \quad (5)$$

The mean SO_4^{2-} value of effluent in GU and GP were 22 ± 8 and 19 ± 9 mg/L, respectively. The lack of carbon sources in GU and GP hindered the conversion of SO_4^{2-}. The lower SO_4^{2-} value in GP than that in GU was possibly caused by plant uptake of SO_4^{2-}.

Removal of Cu, Zn and Pb in OPSU, OPSP, GU and GP

The removal performance of Cu in OPSU, OPSP, GU and GP were shown in Figure 3. The highest mean removal of Cu was in OPSP at 99.80 ± 0.18%. This was followed by OPSU, GU and GP at 99.35 ± 0.93%, 99.28 ± 0.56% and 97.43 ± 2.06%, accordingly. Based on the previously mentioned pH and SO_4^{2-} values, the removal of Cu in OPSU and OPSP could be via sulphide precipitation (Chen *et al*., 2021a). The presence of plants in OPSP further improved Cu removal by plant uptake (Chen *et al*., 2021b).

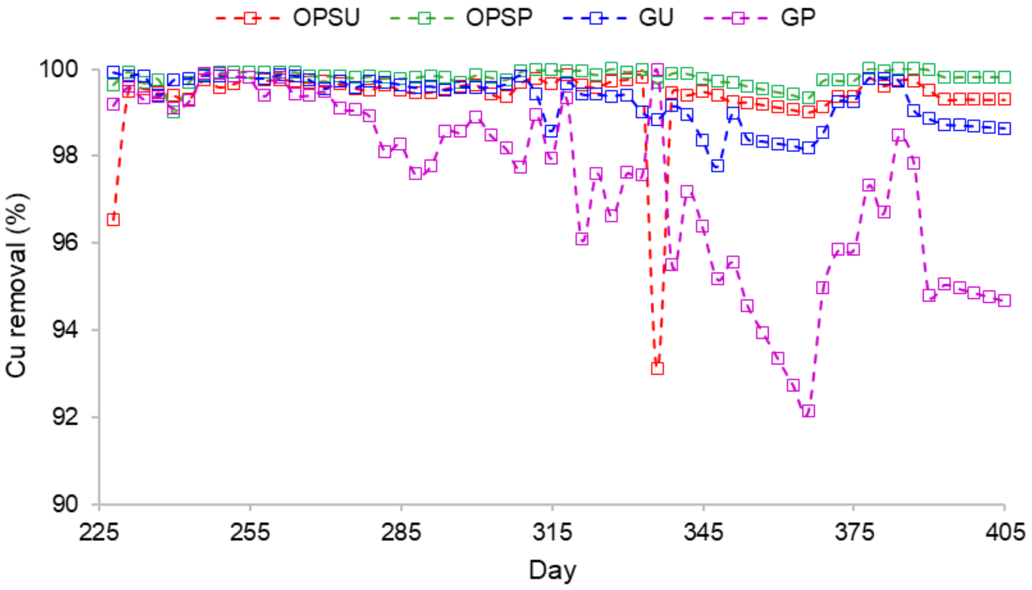

Figure 3. Removal of Cu in OPSU, OPSP, GU and GP

The removal of Cu in GU and GP possibly was caused by carbonate precipitation (Eqs. 6 – 9) (Hua and Haynes, 2016). The higher removal of Cu in OPSU and OPSP could be caused by the presence of organic matter, leading to Cu removal by complexation (Zhang *et al*., 2023). It is interesting to note that the presence of plants in GP decreased the Cu removal. The oxygen released from the plant root in GP could have caused adverse effects on the carbonate precipitation of Cu (Yang *et al*., 2010).

$$CaCO_3\ (s) + 2H^+\ (aq) \rightarrow Ca^{2+}\ (aq) + H_2CO_3\ (aq) \quad (6)$$

$$CaCO_3\ (s) + H_2CO_3\ (aq) \rightarrow Ca^{2+}\ (aq) + 2HCO_3^-\ (aq) \quad (7)$$

$$2HCO_3^-\ (aq) \rightarrow 2H^+\ (aq) + 2CO_3^{2-}\ (aq) \quad (8)$$

$$CO_3^{2-}\ (aq) + M^{2+}\ (aq) \rightarrow MCO_3\ (s) \quad (9)$$

The mean removal of Zn in OPSU, OPSP, GU and GP were 98.56 ± 0.88%, 99.75 ± 0.13%, 83.73 ± 12.42% and 78.36 ± 12.67%, respectively (Figure 4). Similar with Cu, the removal of Zn in OPSU and OPSP were caused by sulphide precipitation as well as plant uptake in OPSP (Chen *et al*., 2021a). Likewise, the decrease in pH buffering capacity of GU and GP media caused decline in Zn removal. The carbonate precipitation of Zn in GP was also possibly affected by the oxygen released from the plant root, which caused the remobilization of Zn carbonate (Yang *et al*., 2010).

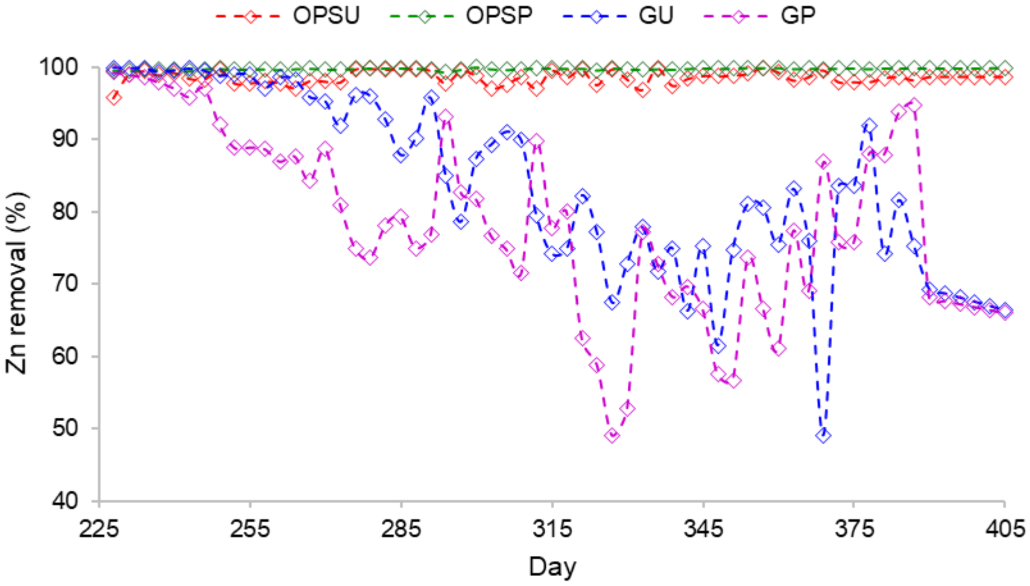

Figure 4. Removal of Zn in OPSU, OPSP, GU and GP

On the contrary, the mean removal of Pb in OPSU, OPSP, GU and GP were almost 100% (Figure 5). The mean removal of Pb in OPSU, OPSP, GU and GP were 99.43 ± 1.07%, 99.66 ± 0.32%, 99.50 ± 0.68% and 99.43 ± 0.58%, accordingly. The removal of Pb in OPSU, OPSP, GU and GP possibly were caused by precipitation (Bavandpour *et al*., 2018; Lizama Allende *et al*., 2011).

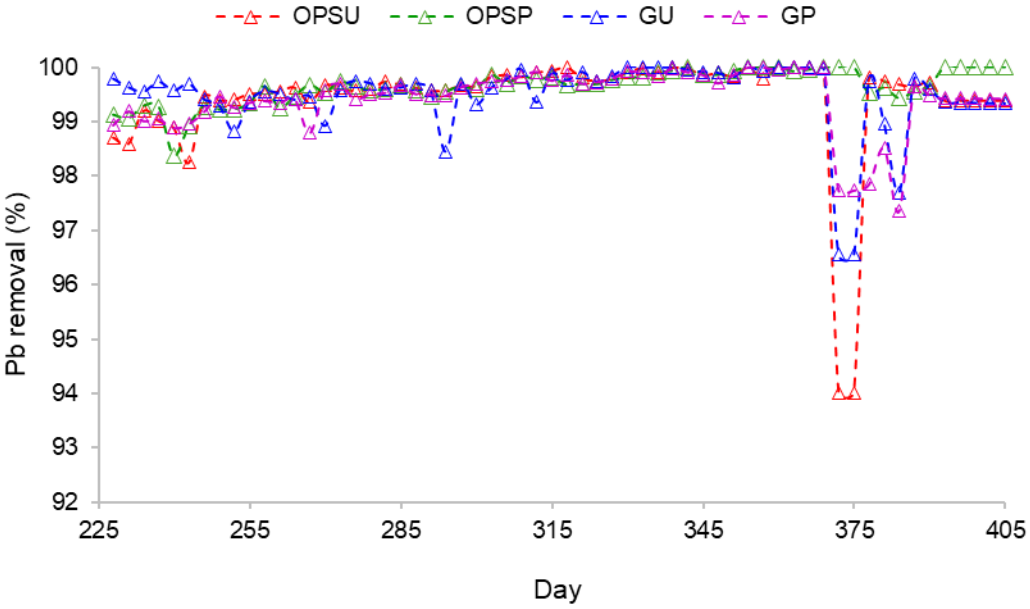

Figure 5. Removal of Pb in OPSU, OPSP, GU and GP

CONCLUSION

In this paper, it was found that the OPS as alternative constructed wetland media performed better than the conventional G media in Cu, Zn and Pb removal, both in the absence and presence of *Typha angustifolia*. The mean pH in OPSU and OPSP were favorable for metal sulphide precipitation whereas in GU and GP, the mean pH was suggesting possible occurrence of metal carbonate precipitation. Further studies on the changes of OPS and G media physicochemical properties after treatment of heavy metal as well as the Cu, Zn and Pb mass balance analysis in OPSU, OPSP, GU and

GP should be conducted to further understand the removal pathway of Cu, Zn and Pb in OPSU, OPSP, GU and GP as well as the role of *Typha angustifolia* towards the heavy metal removal pathway.

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