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Constructed Wetland with Rice Husk Substrate as Phytotechnology Treatment for Sustainable Batik Industry in Indonesia

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Abstract. This study aims to determine the vegetation efficiency of *Canna indica* plants and rice husk as a VFCW substrate in the batik wastewater treatment process. The VFCW pilot is made of acrylic with a length, width and height of 0.5 x 0.5 x 0.5 m respectively. Rice husk is filled as a substrate to a depth of 0.3 m. *Canna indica* is cultivated and acclimatized then be planted in VFCW with 6 plants/units. Batik liquid waste flows with a hydraulic loading rate of 0.15 m³/d and variations in hydraulic retention time for 3 days, 7 days, 15 days, 21 days, and 30 days using an intermittent system. The results showed *Canna indica* proved to have a significant effect ($p < 0.05$) on the removal of all parameters consisting of pH, BOD, COD, TSS, ammonia, and heavy metals Cr. Optimal efficiency for all parameters occurred at 21 days HRT which obtained TSS removal values of 91.25%, BOD₅ of 91.82%, COD of 89.15%, ammonia of 96.2%, and heavy metals Cr of 81.8%. Effluents produced through the construction of VFCW with rice husks as a substrate and vegetation of *Canna indica* are able to meet the standards set out in East Java Governor Regulation No. 72/2013.

1. Introduction

Small industries play an important role in economic development especially in developing countries including Indonesia. The important role is realized in the form of the source of employment creation, potential contribution to improving income distribution, poverty reduction, industry and rural economy, development of entrepreneurship and export growth. At present, the number of small industries in Indonesia is 97% of the total national industry which contributes 58% to the total Gross Domestic Product (GDP). This shows that more than half of the Indonesian economy has been supported by small industries so it is not surprising that small industries are the backbone of the Indonesian economy [1, 2].

The batik industry is one of the potential small textile industries in Indonesia. Batik is a variety of sheet-shaped textile fabrics originating from Indonesian cultural roots. In addition, batik has also been acknowledged by the United Nations Educational, Scientific, and Cultural Organization (UNESCO) as "Intangible World Heritage" in 2nd October 2009 [2]. This determination makes the number of batik small industries continue to grow. The batik industry in carrying out its production process uses a lot of water which 80% is disposed of as waste [3]. The waste contains a high concentration of coloring substances, biological oxygen demand (BOD), chemical oxygen demand (COD), and some heavy metals [4]. The waste generated from these centers will not only pollute the waters but will also have an impact on water scarcity and ecosystem degradation. This impact in particular, will threaten

developing countries which mostly do not have the infrastructure to treat wastewater [5]. Therefore, sustainability is still a challenge in the development of the batik industry in the future.

The development of easy and inexpensive wastewater treatment technology is one of the strategies to support the sustainability of the batik industry in developing countries. Phyto-treatment is one of the green technology candidates that meet both aspects so that it is very potential to be developed [4] [6] [7]. Constructed wetlands (CWs) are one type of Phyto-treatment that has been proven to be efficient, low cost and low maintenance in treating various types of liquid waste [8] [9]. CWs are designed as natural conditions involving wetland plants, substrate media and a group of microorganisms that help treat wastewater [10]. Vertical flow constructed wetlands (VFCW) is one type of CWs which is quite popular because of its wide use in treating various wastewater and not requiring large areas of land. VFCW has good efficiency in reducing total suspended solids (TSS, 90%), chemical oxygen demand (COD, 90%) and ammonia (NH₄-N, 90%). However, the ability of VFCW to reduce phosphate (PO₄-P) is still limited, which ranges from 20-30% [11,12].

The success of VFCW has been studied by several previous studies in treating domestic and industrial waste. One type of industrial waste that can be processed by VFCW is tannery wastewater. The use of local Indian plants, namely Napier Bajra Hybrid grass was able to produce efficiency of decreasing as much as 99% muddiness, 93% TSS, 94% BOD, 82% COD, 88% as Nitrate, 63% as phosphates, and 60% as TN [13]. Based on various previous studies it can be concluded that in addition to the type of wastewater, plant species and substrates are factors that determine the efficiency of the removal of various types of pollutants in wastewater using VFCW. One type of local plant that is widely available is *Canna indica*. *Canna indica* is able to withstand various types of waste, has good removal efficiency, and has an interest that increases the aesthetic value of constructed wetland. This efficiency can be increased using alternative substrates as wetland media that favor the retention of this compound [14]. The Substrate is a medium for biofilm formation by microorganisms that play a role in treating wastewater. One type of material that can be used as a substrate is rice husk. During this time, the rice husk has not been used optimally or has only been dumped as waste.

The application of CWs in processing various domestic and industrial wastes has been extensively tested but this is not the case in treating wastewater in the batik industry. The batik industry is only owned by Indonesia and Malaysia. The different steps of the batik making process and the use of coloring materials in it make the characteristics of industrial wastewater different. The effectiveness study of CWs in testing the liquid waste of the batik industry is a manifestation of Indonesia's contribution in achieving the Sustainable Development Goals (SDGs) established by the United Nations (UN) specifically in the recovery of natural ecosystems such as water management that relate to water quality, supply of water and also water security [15]. This study aims to determine the vegetation efficiency of *Canna indica* plants and rice husk as a VFCW substrate in the batik wastewater treatment process.

2. Methodology

2.1 Characteristics of batik wastewater

The process of making batik takes several steps where it produces different characteristics of wastewater. In this study, wastewater which is used from the pelorodan step because it has more complex characteristics of pollutants. The wastewater samples from the batik industry were obtained from one of the batik industries in Surabaya. The batik wastewater samples were analyzed to determine the initial characteristics before processing using VFCW. The procedure used to analyze the initial characteristics of batik wastewater refers to Standard Methods [16]. Test results on the initial characteristics of batik wastewater are shown in Table 1.

Table 1. Initial characteristic of batik wastewater

Parameter	Unit	Result	Standard (*)
pH	-	10.8	6-9
TSS	mg/L	480	50
COD	mg/L O ₂	424	60
BOD	mg/L O ₂	526	150
Total ammonia	mg/L NH ₃ -N	126.4	8
Total chromium	mg/L Cr	2.2	1

* East Java Governor's Act on Standard of Textile Industry Wastewater No. 72/ 2013

2.2 Wetland construction

VFCW units are made of acrylic with a length, width and depth of 0.5 x 0.5 x 0.5 m or with an effective volume of 0.125 m³. VFCW is applied to treat batik wastewater because it is able to achieve a high oxygen transfer rate so it is efficient in treating ammonia content in batik wastewater [17,18]. Batik wastewater will initially inundate the surface of the wetland and then enter into the substrate with a gravity system [19]. This condition will increase aeration that supports biological processing in the system [20]. Therefore, batik wastewater flows intermittently in the cycle of filling and drying on substrate media [21].

2.3 Plants and media

Plants in CWs play a very important role in purifying the wastewater. The important role of the plant is held by the root which functions as an inherent area of microbes which makes it possible to grow and develop to be able to absorb and decompose wastewater. In addition, plant vegetation provides additional benefits because it is a wildlife habitat, increases aesthetic value, and creates a greener environment [10]. The plants used in this study were *Canna indica*.

The selection of *Canna indica* is motivated by its abundant availability in the majority of tropical regions, has a high growth rate, has good nutrient removal abilities, and has beautiful flowers. *Canna indica* has been known as a phytoremediation plant in a constructed wetland. This is due to having a greater number of roots, a higher rate of development of the root system, larger root biomass, and a wider area of surface than other plants. In addition, these plants have a high tolerance for various types of pollutants and have a long root life [22]. *Canna indica* before being used in wetlands must be cultivated first for 1 month with rhizomes at a rate of 6 plants/units. Then the plants were acclimatized for 1 month by flowing diluted wastewater into VFCW (50% tap water: 50% primary treated sewage effluent).

The substrate in CWs serves as a growing medium for macrophyte plants, supporting chemical and biochemical transformation, where pollutants are lowered and where wastewater is moved [8,14,23]. In addition, the substrate functions as the layer attachment surface [6]. The substrate used in this study is rice husk (RH). RH is classified as organic material [6,24]. RH was obtained from one of the farms in Pasuruan and was directly used in the cells. Rice husk contains 75% organic substances (cellulose, lignin, hemicelluloses), 15% minerals, and 10% water and microelements [25]. RH is washed first using tap water before being used as VFCW media. The depth of the growth media was 0.3 m and the minimum level was 5 cm below the surface of the media.

2.4 Experimental procedure

This research was conducted in the dry season where VFCWs pilots were placed in the outdoor to obtain natural conditions such as sunlight and wind. In this study, simple settling basins were used as the pretreatment steps before batik wastewater flows into VFCW. This is done to prevent porous media clogging and reduce the organic load that may be caused by suspended solids [26]. Furthermore, batik liquid waste is flowed into the wetland bed using a peristaltic pump to maintain a constant water level in the distributor inlet and adjust the inflow rate.

Batik wastewater enters the wetland bed where the flooding and draining of cycles were conducted by drainage system under reactors. Flooding is done by filling the wetland bed through drainage valve closure while draining is done by opening the drainage valve until the wetland bed is completely dry [27]. The hydraulic retention time is varied 3 days, 7 days, 15 days, 21 days, and 30 days with the hydraulic loading rate is 0.15 m³ / d. The performance of the VFCW was observed under the presence and absence of vegetation. The effluent measurement process is replicated 3 times at each hydraulic retention time.

The effluent has then analyzed parameter pH, TSS, COD, BOD, total ammonia, and total chromium. All parameter analysis effluent refers to standard methods. pH was measured in the field using a portable meter (Hanna HI9811-5, Hanna Instruments, Romania), the COD was measured by a closed reflux titrimetric method, the BOD were measured according to the 5-d BOD test, ammonia nitrogen was measured by titration with HCl, the TSS were measured after evaporating the sample to dryness at 103°–105°C [3], and total chromium were measured using an Agilent 7500c ICP-MS in helium collision mode according to EPA Method 200.8 [4, 5].

2.2 Data analysis

In order to investigate statistically significant differences, a one-way between-groups analysis of variance (ANOVA) at 95% significance level was used to examine the performance of the various filters. All statistical data were analyzed using SPSS 17.0. All statistical analyses were performed using SPSS 17.0 for Windows.

3. Results and Discussion

Temperature is a very important environmental factor in constructed wetland operations not only affecting plant growth, the process of exchange of substances (metabolism), but also the root exudates of plants [30]. In this study, the measurement process is carried out every day in the inlet and outlet section where a temperature range of 28–36°C is obtained. Observations show that at this temperature *Canna indica* can grow and develop well. This can be seen where the leaves of the plant remain green, stand tall and are capable of flowering.

In addition to temperature, observations are also made to pH. pH in wastewater is an important factor that influences wetland performance especially in the removal of nitrogen and organic matter. The pH value indicates the concentration of H⁺ and OH⁻ ions in wastewater. The higher the H⁺ ion indicates that the waste is acidic. The higher OH⁻ ion indicates that the waste is alkaline. Observation of pH is carried out every day with a value between 7.5–9.8. The results of the observations show that the longer the exposure time, the pH is towards neutral. This shows that the wastewater treatment process can affect the pH value of treated water to be lower. This situation shows that *Canna indica* plants are a type of plant that secretes H⁺ ions. While the process of respiration of plants produces CO₂ which can increase the pH value. This happens because the CO₂ released by plants from respiration results is reused for photosynthesis so that it will shift the balance to the right, which means there is a reduction in H⁺ ions in batik wastewater.

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The pH value as shown in Figure 1 obtained values of pH fluctuations on HRT 3 days, 7 days, 14

days, 21 days and 30 days even though on the last day the standard effluent determined for VFCW was planted with *Canna indica*. VFCW which is not planted with *Canna indica* so that the last HRT does not meet the effluent standards set by the government for pH values of 6.5-7.5 where bacteria can still grow and survive [5]. Vegetation plays an important and significant ($P < 0.05$) role in stabilizing pH. The pH value that is close to neutral is due to decreasing concentrations of BOD and COD and are close to stable. Water pH greatly affects the biochemical processes in water. The increase in pH is due to photosynthesis, denitrification, organic breakdown and sulfate reduction [31, 32, 33].

The pH value is in the highest rate for denitrification processes and degradation processes of organic matter as reported by [34]. This is because of the high sensitivity of bacteria responsible for the formation of methane gas in the system. An increase in the pH value is also likely due to the due to the alkaline nature of RH contributed by carbonates, bicarbonates. The prime reason for high COD removal is due to filtering, settling and adsorption process contributed due to RHA. The removal of the pollutants by RH depended on various factors such as adsorption, precipitation, filtration, sedimentation, microbial degradation, and plant uptake. Moreover, the high surface area of the RH may be a key factor to facilitate pollutant degradation [35].

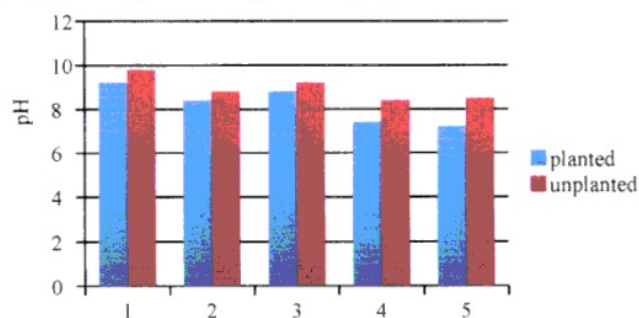


Figure 1. The pH value to HRT on a variety of conditions is planted with *Canna indica* and unplanted

The BOD/COD ratio is a parameter that indicates the level of biological degradability. A categorized waste is easily broken down if it has a BOD/COD ratio value greater than 0.5 [36]. The BOD/COD ratio of batik wastewater 1.24 so that it can be concluded that wastewater batik is classified as easily biodegradable. The results show that BOD efficiency removal in all conditions (planted and unplanted) is higher than COD. Nevertheless, the decrease in BOD and COD occurred along with HRT as shown in Figure 2 and Figure 3. Vegetation plays an important and significant ($P < 0.05$) role in BOD and COD removal. The average percent removal for BOD on planted beds is 69.84% while on the unplanted bed is 48.74% while for percent removal COD is 61.22% on planted beds while on the unplanted bed is 40.18%. Removal of BOD and COD is influenced by the activity of the organism and the ability of *Canna indica* in absorbing nutrients, where the longer the exposure time, the more organic material will be set aside through a biodegradation mechanism.

Organic materials contained in batik wastewater become nutrients for microbes that will break it down into a simpler substance. Increased BOD and COD removal continues to increase with time due to the intermittent flow mechanism, cycle drying provides an opportunity for plants to restore their absorbing ability [4, 27]. The results of previous studies state that media types do not have a significant effect on BOD removal in both vegetation conditions and the type of media used. Lower values are indicated at 86.2% and 80% reported by [37, 38]. The degradation process by microbes begins when there is contact between organic material and biofilm layers formed in submerged plant stems, roots, surrounding soil or media via diffusion processes. *Canna indica* is a medium of oxygen aerobic and conveys degradation through the rhizosphere [39, 40]. The results of this study have similarities with previous studies, which resulted in higher BOD removal in beds with plants which

amounted to (72.4-78.9%) compared to beds without plants [41]. High removal values are likely caused by deposition and filtration processes that eliminate quickly settleable organic compounds [42].

BOD removal results after 21 days did not show a significant effect due to planted beds experiencing outperform control, a condition where *Canna indica* stimulates the microbial community density and activity by providing root surfaces for microbial growth, and a source of carbon compounds through root exudates [43]. This result is also not much different from previous studies where maximum BOD removal occurred at 10-14 days retention time with values reaching 90.0% [44, 45]. HRT is one of the factors that can be controlled within CWs. For example, BOD removal efficiency is obtained when HRT is in the range of 1-3 days [46]. Longer HRT shows a higher allowance efficiency because more optimal contact occurs but is a dilemma because it affects the needs of the larger area. This condition is an obstacle in areas that have limited land [47].

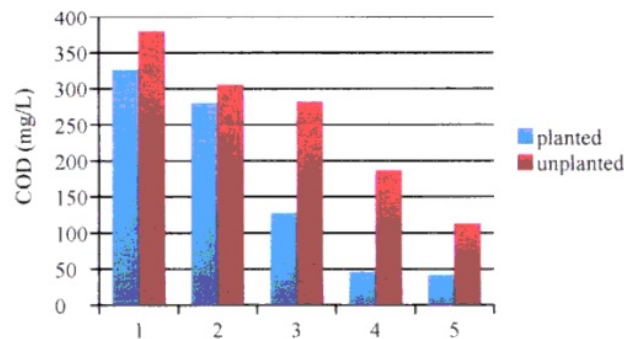
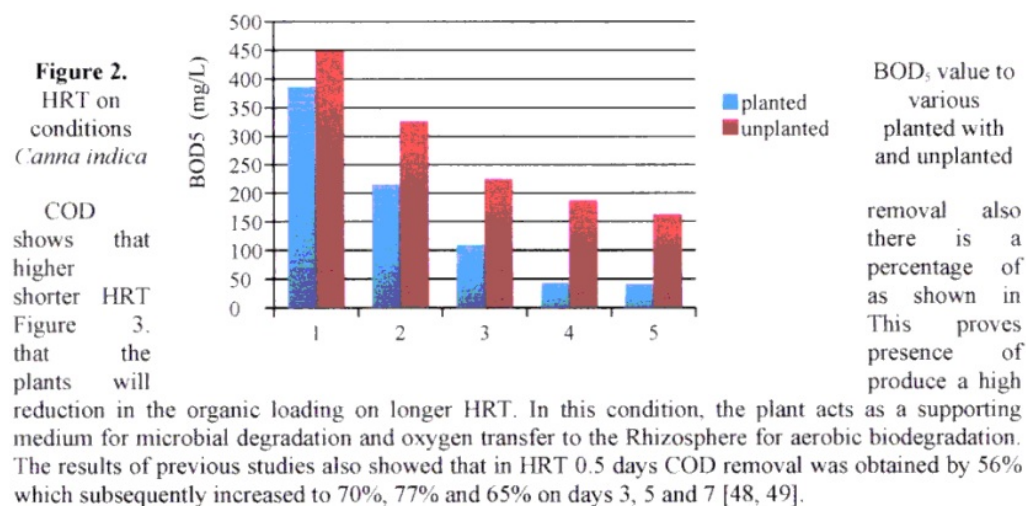


Figure 3. COD value to HRT on various conditions planted with *Canna indica* and unplanted

The TSS removal process showed that percent TSS removal on planted beds was 61% higher than unplanted beds as 48% as shown in Figure 4. The average percent TSS removal in this study was 1.3 times higher on planted beds than the unplanted bed. The results of this study are similar to those of Zhao et al. which used alum sludge as a substrate on VFCW [50]. TSS removal occurs mainly through physical processes such as sedimentation and filtration [43, 51]. Filtration occurs because of particle impaction into the root and macrophyte stem or into the substrate on VFCW. In this study, *Canna*

indica has an extensive root system that provides a large surface area, lowers water velocity, and reinforces increasing settling and filtration in the root systems. This is what increases TSS removal efficiency [48]. In this study, VFCW has a longer lifetime, the wastewater batik is deposited first in the settling basins to prevent clogging that occurs in the VFCW substrate [52].

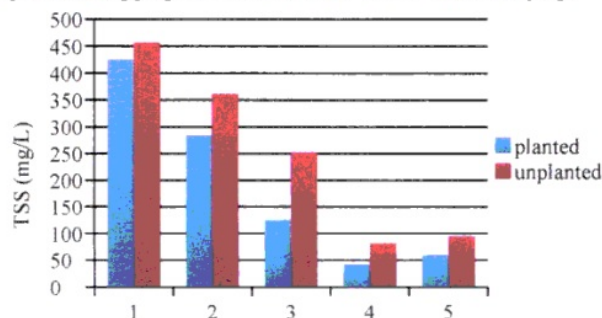


Figure 4. TSS value to HRT on various conditions planted with *Canna indica* and unplanted

Total ammonia nitrogen, the predominant form of nitrogen in influent, is removed in wetlands through a combination of processes, comprising nitrification, denitrification and plant uptake. Nitrification needs an aerobic environment, while denitrification needs an anoxic environment. The role of the plants was observed to be significant in the case of NH_4^+ removal and nitrogen transformation when constructed wetlands were operated at one day hydraulic retention time. The plants were proven to accelerate the process, as a lower concentration of NH_4^+ was observed at the planted cell. The superiority of planted wetland cells in NH_4^+ removal was also reported by other researchers. The nitrification process occurs in the aerobic rhizosphere where atmospheric oxygen is transferred to the root zone through the wetland plant.

The significant difference of NH_4^+ concentration in effluents from cell with 0.5 day hydraulic retention time and one day hydraulic retention time was recorded. This observation revealed that constructed wetlands needed more than 0.5 days fully to nitrify the ammonia loaded to the system. When hydraulic retention time was increased from one day to three and seven days in phase 2, the removal of NH_4^+ was further improved. During phase 2 the concentrations of NH_4^+ decreased by 97% on average which was better than those during phase 1. This finding is in agreement with those obtained by Mayo and Mutamba. They have documented higher NH_4^+ removal for eight days of hydraulic retention time compared to five and three days. For parameters where the influence of hydraulic retention time was found to be significant, the relationship between hydraulic retention time and effluent quality was described by curve estimation analysis [6].

In general, the removal efficiency of NH_4^+ under all tested conditions was low and followed the order: planted beds (32%) > unplanted beds (26%), the vermiculite media (33%) > gravel media (25%) and the batch feeding (36%) > continuous feeding (22%). The relatively high removal rate of NH_4^+ under planted vs. unplanted conditions could be explained by plant uptake and the higher rate of nitrification. This result implies that NH_4^+ uptake by the plant is a minor factor compared to the nitrification process, which is considered the major NH_4^+ removal process. Statistical analysis ($P < 0.05$) showed that NH_4^+ removal was significantly affected by the type of media and mode of feeding in planted beds. The higher efficiency of NH_4^+ removal by RH is possibly due to the higher cation exchange capacity (CEC) [7]. These results are in good agreement than [17] where ammonia nitrogen is removed by 45.3 and 34.8% for planted and unplanted constructed wetlands respectively and [23] where nitrogen is removed by 49.37% through planted sand filter although for the effluent of secondary treated wastewater followed by floating bed. The observed results of this research are in

accordance as [14]. Overall removal found from all treatment beds could be reasoned: vegetation influences interaction between plants-wastewater—microorganism, provide microbial attachment sites, sufficient wastewater resident time, trapping and settlement for wastewater components, the surface area for pollutants adsorption, uptake storage in plants diffusion of oxygen from rhizosphere [8, 24].

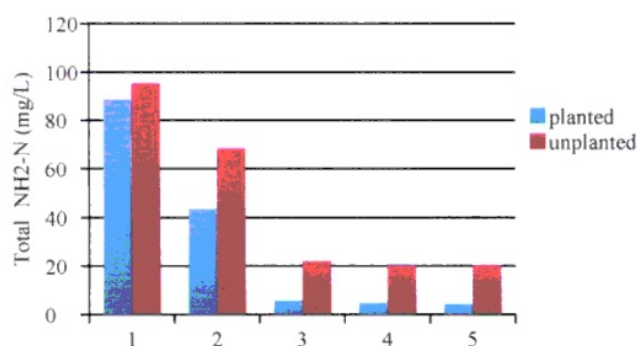


Figure 5. $\text{NH}_2\text{-N}$ value to HRT on variations in conditions planted with *Canna indica* and unplanted

The ammonia removal process in this study also showed a higher value on planted beds which was an average of 76.8% compared to an unplanted bed of 64.1% as shown in Figure 5. Statistical analysis showed that vegetation had a significant effect on TSS removal ($P < 0.05$). Ammonia removal process occurs through adsorption, nitrification, plant uptake, and volatilization. Rice husk is well known in the ammonia removal process [12, 32]. Ammonia adsorption is carried out by rice husk as a substrate of wetland which then becomes a source of nitrogen for plants because it forms the NH_3 -biochar complex [32]. Rice husk pores become microenvironment conditions and favor the growth of microbial communities such as ammonia-oxidizing bacteria [33, 35].

The ability of CWs in processing heavy metals has been proven in several previous studies. In this study, the percent removal of Cr metal averaged 60.9% on the planted bed while the unplanted bed was 15.5% as shown in Figure 6. However, statistical results showed that vegetation did not have a significant effect on Cr removal ($P < 0.05$). This can happen because most heavy metals Cr is retained on the ground, gravel and biofilm and a small portion of taken by plants. The root system in vegetation is able to absorb large amounts of heavy metals. Different percentage removal rates indicate a different level of mobility and bioavailability [21, 35]. This condition shows that metal removal of Cr can occur through various processes such as precipitation, cation exchange with clay soil, adsorption to roots and gravel, bioaccumulation by microorganisms and chemisorption [54, 55]. This study is different from previous studies where unplanted beds showed higher removal values in CWs that used soil as a substrate. This is probably due to the plant uptake, adsorption of Cr on the root surface by chelating agents which reduce the availability of Cr in the cation exchange in planted CWs process. This phenomenon contributes to the efficiency of the removal of high chromium metal. In this study, *Canna indica* is able to accumulate Cr in roots and stems and leaves through extracellular negatively charged sites (COO^-) from the immobilized root cell walls and Cr in the vacuoles of the root cells [56].

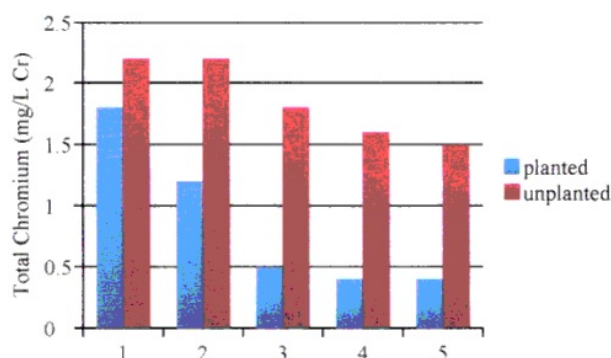


Figure 6. The total value of chromium to HRT in a variety of conditions is planted with *Canna indica* and unplanted

4. Conclusion

The results indicate that VFCW with rice husk as a substrate and *Canna indica* plants proved effective in treating batik wastewater. *Canna indica* proved to have a significant effect ($p < 0.05$) on the removal of all parameters consisting of pH, BOD, COD, TSS, ammonia, and heavy metals Cr. The effectiveness of this system is indicated by a pH value of 7.4 at 21 days HRT which is within the standard range of 6-8 effluents. In addition, the TSS removal value is 91.25% for the 21st HRT day or an overall average of 61%, BOD5 removal is 91.82% at 21 days HRT or the overall average is 69.84%, COD removal is 89.15% in HRT to 21 days or an overall average of 61.22%, ammonia removal of 96.2% at HRT to 21 days or an overall average of 76.8%, and removal of Cr heavy metals at 81.8% at HRT to 21 days or an overall average of 60.9 %. Optimal removal efficiency for all parameters occurred at 21 days HRT because 30 days of HRT did not significantly increase removal efficiency and some even decreased as TSS. This is probably due to the rice husk pores starting to experience clogging. The VFCW construction in this study was able to produce effluent from processed batik wastewater that met effluent standards as stipulated in East Java Governor Regulation No. 72/2013.

5. Acknowledgment

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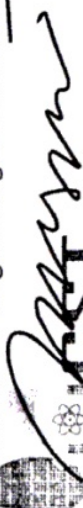
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