Synthesis of Eichhornia crassipes Biochar: Sustainable Efficient Adsorbent for Reducing Cr (VI) Metal Ion

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Abstract. Chromium (Cr) is the most widely used metal in industrial activities and is the most toxic heavy metal ion found in waters. Utilization of Eichhornia crassipes as a Sustainable Efficient Adsorbent of heavy metal Cr (VI) is one of the innovations and alternative technologies that are very beneficial for society and the environment. The synthesis of Eichhornia crassipes biochar in this study employed the hydrothermal method. Hydrothermal temperature variations used in biochar synthesis were 220°C/240°C/260°C/280°C. Eichhornia penetrated biochar with H2O2 to the Cr (VI) metal adsorption capacity. The synthesis of biochar Eichhornia crassipes has the characteristics of combining hydroxyl, carbonyl, carboxyl, and amine functional groups. The surface morphology in the Eichhornia crassipes biochar modification term is rougher and irregular compared to Eichhornia crassipes biochar without modification. The modification of Eichhornia crassipes biochar using H2O2 showed an increase in oxygen composition in terms of modified biochar so that it increased the adsorption capacity of Cr (VI) metal ion. The best value of adsorption capacity in Eichhornia crassipes biochar terms modified with a hydrothermal temperature of 240°C that is equal to 30.2156 mg/g.

1. Introduction

The contamination of water by heavy metals has become a serious problem throughout the world because of the nature of heavy metals which cannot be decomposed naturally, accumulating over time, toxic, and contain cancer-causing substances, which in turn prove to be bad upon living things [1]. Among all, Chromium (Cr) is the most widely used metal in industrial activities and is the most toxic heavy metal found in waters [2]. Contact with a high level of Cr (VI) metal ions can cause epigastric pain, nausea, vomiting, acute diarrhea, and digestive tract and lung cancer [3][4].

Efforts to handle Cr (VI) metal ions waste in waters have been carried out, and adsorption is one of the many choices that were used for absorbing Cr (VI) metal ions from industrial wastewater [5]. The use of adsorbents in the process of adsorption of heavy metals in liquid waste has long been known. This process is actually quite suitable to be developed in Indonesia. One of the reasons why this process is not used by industry is because of the price of adsorbents is quite expensive [6]. The usual adsorbent used is activated carbon produced from the carbonization process of coal, which will require very expensive synthesis costs [7][8]. Higher cost and operational losses such as combustion at high temperature, pore blocking, and hygroscopicity are several examples of the disadvantages of the utilization of activated carbon for decolorization in various industrial adsorption and separation processes, despite its efficient adsorption of the organic compound, that motivate large number of researchers to find cheaper substitutes for activated carbon lately [6]. The manufacture of activated carbon using materials that are easily found at low prices also began to be developed, such as by
utilizing coconut shells, biomass fuel, drying rice, eggshells and mangosteen peels [9]. However, the availability limitations of the basic ingredients of this adsorbent both in quantity and continuity have become a constraint on its own in developing adsorbents. The development of adsorbents by using raw materials at low prices, guaranteed quantity, and continuity, and process that can be easily carried out by the industry itself is certainly interesting to be developed [10].

One of the adsorbents that meet these criteria is the use of biochar based on Eichhornia crassipes. Biochar is a carbon-rich product resulted from thermal decomposition of biomass such as wood, leaves, agricultural waste, solid waste, and abundant natural biomass, with very limited oxygen conditions at low temperatures (<700°C) [7][11]. Due to its high surface area, high porosity, cation exchange capacity, and pH buffering ability as a solid product obtained from the thermochemical conversion of biomass, biochar has been applied to wastewater treatment and remediation of contaminated soil [12]-[16]. Compared to activated carbon, biochar is an inexpensive, more environment-friendly and very potential alternative adsorbent because of its wide application in every area of life [17]-[19]. Eichhornia crassipes is a weed that grows in shallow waters especially in tropical and subtropical regions [20]. The ability of Eichhornia to propagate by absorbing heavy metals, minerals, water nutrition, and organic chemicals results in its rapid growth [20][21]. This excessive growth leads to serious environmental problems such as closed surface, siltation, and reduced supply of oxygen on the water which causes death to fishes and agricultural plant irrigation systems problems [22]. Besides having a negative impact caused by Eichhornia crassipes, naturally, the water hyacinth root has the ability to absorb heavy metal waste such as Cu, Cd, Ni, Pb, and Zn [23]. Hasan et al. (2010) reported that water Eichhornia crassipes biomass (WHB) has shown high potential for removing hexavalent chromium from aqueous solutions with the Box-Behnken RSM design where the R-value is 99.8% [24]. Moreover, the example of the removal of metal ions like chromium, zinc, and copper from the textile effluent collected from Lahore district, Pakistan shows the ability of Eichhornia crassipes as reported by Mahmood et al. (2005) [25].

Utilization of Eichhornia crassipes as biochar capable of adsorbing heavy metals is an innovative and technological alternative that is very beneficial for society and the environment [26]. To increase the effectiveness of biochar based on Eichhornia crassipes, this biochar is activated by H$_2$O$_2$ activator. According to Yingwen Xue, et al. (2012), biochar that has been activated by H$_2$O$_2$ can activate and enhance the ability of biochar to adsorb heavy metals [18]. Liquid phase oxidation with H$_2$O$_2$ significantly increases acidic oxygen-containing groups on the surface of activated carbon without causing significant physical changes [27][28]. Based on the description, research will be conducted on the biochar synthesis based on Eichhornia crassipes with hydrogen peroxide activator (H$_2$O$_2$) as a sustainable efficient adsorbent, and the adsorption capacity test for Cr (VI) ions.

2. Methodology

2.1. Materials

The materials used in this study were the leaf stalks of Eichhornia crassipes taken from the relocation area of the Johar market Semarang, Central Java Indonesia, deionized water, 10% H$_2$O$_2$, K$_2$Cr$_2$O$_7$, NaOH, HCl, and distilled water.

2.2. Preparation and modification of biochars

Synthesis of Eichhornia crassipes biochar was carried out by the Hydrothermal method. The function of combustion using this hydrothermal method is to increase carbon content in biomass and convert biomass to biochar [29]. 25 g of dried stalks of Eichhornia crassipes together with 200 mL of deionized water in the autoclave reactor was heated at 220°, 240°, 260°, and 280°C for 1 hour. The reactor is then cooled at room temperature and the biochar product is collected. The resulting biochar is then filtered and dried in an oven at 65°C for 24 hours [30]. To make modified biochar Eichhornia crassipes, about 3 g Biochar is mixed with 20 ml of 10% H$_2$O$_2$ solution and allowed to stand for 2 hours at room temperature. The H$_2$O$_2$ can oxidize carbonated surfaces and improve oxygen-containing functional groups, especially in carboxyl groups on biochar surfaces [27]. In addition, H$_2$O$_2$ can also function as an oxidation reagent to modify or activate Biochar so as to produce an effective adsorbent
in removing heavy metals. The biochar product that has been soaked for 2 hours is rinsed with deionized water and dried in an oven at 80°C [18].

2.3. Characterization of biochars
To detect functional groups and active sides of biochar, Fourier Transform Infrared Spectroscopy (FTIR, Bruker) at wavelengths of 400–4000 cm\(^{-1}\) with a spectrum separation of 2 cm\(^{-1}\), at 20°C at KBr pellet method is used [31]. Furthermore, Scanning Electron Microscope (SEM) analysis is used to determine the surface morphology of biochar. The adsorbent surface morphology analysis was then determined using the Scanning Electron Microscope SEM (FEIQuanta 200, FEI, USA). Lastly, Energy Dispersive Spectroscopy (EDS) analysis was used to determine the elemental composition of biochar [31].

2.4 Adsorption experiments
Synthetic wastewater is prepared by making Cr(VI) mother liquor. 1000 ppm Cr (VI) mother liquor was prepared by dissolving as much as 2,828 g of anhydrous potassium dichromate (K\(_2\)Cr\(_2\)O\(_7\)) and 1.5 ml of HNO\(_3\) (1M) in 1 liter of deionized water. The mother liquor is then diluted according to the concentration needed when testing the adsorption capacity [10]. In this study, the concentration of Cr (VI) of 100 ppm was used. Adsorption was carried out on 25 ml of artificial waste with a variation of contact time for 1 hour, reaction pH 5 and biochar mass was 0.08 g. Analysis of Cr (VI) metal ions concentrations was carried out using AAS. The adsorption capacity (mg/g) of the biochar for the adsorption of Cr (VI) metal ions can be calculated by the following equation [32]:

\[
Q_e = \frac{(C_o - C_e)V}{m}
\]

Where \(Q_e\) is the adsorption capacity (mg/g), \(C_o\) and \(C_e\) are the initial concentration and equilibrium (mg/L) of the adsorbate in the solution, \(V\) is the volume of the solution (L), and \(m\) is the mass of the adsorbent (g). The percentage of metal removal was calculated using the following equation [33]:

\[
\text{Adsorption (\%)} = \frac{(C_o-C_e)}{C_o} \times 100
\]

3. Result and Discussion
Biochar synthesis results were then analyzed using Fourier transform infra-red (FTIR). The FTIR analysis aims to determine the existence of functional groups in biochar. The biochar FTIR spectra results based on biochar comparison without modification and modification can be seen in Figure 1.

![FTIR Spectra](image)

**Figure 1.** FTIR spectral analysis of Eichhornia crassipes biochar (ECB) without modification with hydrothermal temperature 220°C and Eichhornia crassipes biochar modified (ECBM) with hydrothermal temperature 220°C, 240°C, 260°C, 280°C
Subsequent to hydrothermal treatment, the biochar held substantial oxygen-containing functional groups. Based on Figure 1, the stretching of hydroxyl (O-H) functional groups is due to the dehydration of the holocellulose at the intensity of the peak at 3600-3000 cm\(^{-1}\) [13]. The temperature of the hydrothermal is increasing along with the stretching of –C–H of the methylene groups, thus explained the approximately 2921 cm\(^{-1}\) peak point [34]. In line with the previous findings, the aromatization is reflected by the vibration of the stretching of -C==C aliphatic carbon (1650–1450 cm\(^{-1}\), C==C) that is linked to both peak at 1517 cm\(^{-1}\) and 1610 cm\(^{-1}\) [35], therefore, the aromatic structures are possible to be formed from (Hemi-)cellulose under both the hydrothermal and the non-hydrous circumstances. Moreover, the stretching of -C-O was linked respectively to phenolic, and alcoholic suitably at peak 1023 cm\(^{-1}\) and 1059 cm\(^{-1}\) [36]. The carboxylic groups were linked suitably to the bending of -O-H at 1205 cm\(^{-1}\) peak point. [37]. The N–H group corresponds to a peak at 1418 cm\(^{-1}\) [10]. Furthermore, a substantial amount of quantity of acidic oxygen-containing functional groups (OFG), in an instance, hydroxyl and carboxyl existence are confirmed by all. It has to be known also that the chemical reaction, such as surface complexation and ion exchange, is impacted significantly by the key factors that are the surface OFGs [38]. Therefore, the attractive property of the biochar could be greatly improved by a substantial amount of quantity of surface OFGs. Based on the spectrum of FTIR results in figure 1 there are various hydroxy, carbonyl, amine, and carboxyl functional groups on the biochar surface. Compounds containing groups such as hydroxyl, carboxyl, amino, carboxylate, and alkoxy have a great affinity for heavy metals [8][3].

Biochar synthesis results were further characterized using Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS). The results of SEM and EDS analysis based on biochar comparison without modification and modification at 240 can be seen in Figure 2.

![Figure 2. SEM of ECB 240°C (A), (B) ECBM 240°C and spectra EDS of ECB 240°C (C), ECBM 240°C (D)](image)

Based on Figure 2 SEM ECB and ECBM (A and B) synthesized with 240°C hydrothermal temperatures at magnification 2000x show the surface morphology of the ECBM is rougher and irregular than the ECB. This is caused by modifications using \(\text{H}_2\text{O}_2\). The results of EDS analysis in figures 3.C and 3.D show the composition of oxygen elements in the modified biochar has increased from 54.24 to 74.65%. Based on the increase in the composition of these elements it can be said that
biochar was successfully modified with H$_2$O$_2$. A number of the increasing oxygen-containing functional groups (particularly –COOH) are in line with the findings from another study that shows part of the carbon in biochars was oxidized by H$_2$O$_2$ treatment [39]. This result also supported by research done by Xue, et al. (2012) which states that carbon in PHHC was oxidized by the H$_2$O$_2$ resulting in the calculated higher oxygen content of the mPHHC (44%) versus that of PHHC (37%) [18]. This is also in line with the findings of previous studies that H$_2$O$_2$ treatment is able to oxidize carbonized surfaces and increase the oxygen-containing surface function groups, carboxyl group in particular [27].

The results of SEM and EDS analysis of Eichhornia crassipes biochar modified with a hydrothermal temperature of 240°C (ECBM240) before and after the adsorption of Cr (VI) metal ions can be seen in Figure 3.

Based on the results of the EDS analysis in figure 3, the adsorption of Cr (VI) metal ions was successfully confirmed by the Cr (VI) peak shown on the EDS spectra image (figure 3.D). The EDS image also shows the loss of Mg element composition in ECBM240 after adsorption. This is due to the mechanism of ion exchange between Mg$^2+$ and Cr (VI). In addition, the EDS figure 3.C and 3.D showed a decrease in the composition of the oxygen element from 74.65 to 68.60% due to the presence of Cr (VI) metal ions bond with oxygen-containing functional groups. The adsorption capacity test carried out using a biochar mass of 0.08 g, contact time of stirring for 60 minutes with a solution concentration of 100 mg/L and pH of solution 6. The results of the analysis of the adsorption capacity test can be seen in Table 1.
Table 1. Comparison Cr (VI) Metal Ion Adsorption Capacity Test of unmodified biochar (ECB) and modified biochar (ECMB)

| Adsorbent | Hydrothermal Temperature (°C) | Concentration Post Adsorption (mg/L) | Adsorption Capacity (mg/g) | % Cr Metal Reduction |
|-----------|------------------------------|-------------------------------------|---------------------------|---------------------|
| ACB220    | 220                          | 43,234                              | 17,7394                   | 56.77               |
| ACB240    | 240                          | 38,782                              | 19,1306                   | 61.22               |
| ACB260    | 260                          | 36,532                              | 19,8338                   | 63.47               |
| ACB280    | 280                          | 35,231                              | 20,2403                   | 64.77               |
| ACBM220   | 220                          | 8,532                               | 28,5838                   | 91.47               |
| ACBM240   | 240                          | 3,31                                | 30,2156                   | 96.69               |
| ACBM260   | 260                          | 3,994                               | 30,0019                   | 96.01               |
| ACBM280   | 280                          | 4,173                               | 29,9459                   | 95.83               |

ACB : Eichhornia crassipes biochar  
ACBM : Eichhornia crassipes biochar Modifikasi

Table 1 shows that the hydrothermal temperature of Eichhornia crassipes biochar can affect the value of adsorption capacity. Based on the data obtained, the best value of adsorption capacity in Eichhornia crassipes biochar without modification (ECB) is shown at temperature 280°C with a value of 19.1306 mg/g. The best adsorption capacity on Eichhornia crassipes biochar modified (ACBM) was shown at temperature 240°C with a value of 30.2156 mg/g. In addition, based on the results of the adsorption capacity test, the Eichhornia crassipes biochar modified (ACBM) has a better ability to absorb Cr (VI) metal ions compared to ACBM without modification (ACB). The amount of H₂O₂ modified biochar adsorption capacity is caused by the number of oxygen-containing groups on the surface of the modified biochar compared to biochar without H₂O₂ modification which was observed from the results of EDS measurements. Acid pretreatment and biochar chemical oxidation generally increase metal adsorption. H₂O₂ oxidation increases the number of carboxyl groups on the biochar surface and provides additional cation exchange sites for surface complexation of Pb (II) [18] and Hg [28], which are comparable or even superior to commercial activated carbon. Modified biochar H₂O₂ showed an increase in absorption of Pb (II) with an absorption capacity of 22.8 mg g⁻¹, which is more than 20 times that of unmodified biochar (0.88 mg g⁻1 due to an increase in oxygen-containing functional groups). Respectively, the oxygen-containing groups were ascribed to alcoholic, phenolic, and carboxylic groups. Furthermore, these groups on the surface of biochars were related to the hydrolysis and decomposition of major bio-polymer components in Eichhornia crassipes, and it also takes a significant part in the interactions with the Cr(VI) particles in solutions. This is in line with research conducted by Jian et al, 2018 which states that the large copper metal adsorption capacity is due to a large number of oxygen-containing groups on the surface of rice husk biochar [34]. Thus the modification treatment using H₂O₂ on Eichhornia crassipes biochar can activate biochar and improve the ability of biochar to absorb heavy metals such as Cr (VI). Different examinations have demonstrated that the removal of heavy metals by biochars, including biochar, is straightforwardly related to the measure of oxygen-containing functional groups [36][40][41]. These groups can also act as acids or bases, showing properties of ion exchange and coordination on activated carbon [27]. In this study, the increase of carboxyl surface functional groups mainly related to the enhanced chromium removal by the H₂O₂-modified biochar. Moreover, The literature also stated that the metal can react to carboxyl groups on carbonized surfaces in order to form bound complexes [21][42].

4. Conclusion

Synthesized biochar Eichhornia crassipes contains characteristics of hydroxyl, carbonyl, carboxyl, and amine functional groups. The surface morphology of the modified biochar Eichhornia crassipes is rougher and irregular compared to Eichhornia crassipes biochar without modification. Modifications using H₂O₂ on Eichhornia crassipes biochar showed an increase in the composition of the oxygen element in the modified biochar. It was discovered that H₂O₂ modification can initiate biochar created from Eichhornia crassipes and significantly improve its capacity to remove Cr(VI) metal from water.
The best adsorption capacity value is in the modified biochar Eichhornia crassipes with a hydrothermal temperature of 240°C, the value is 30.2156 mg / g.

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