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Removal of Cadmium (II) by Adsorption Using Water Hyacinth (*Eichhornia crassipes*) Dried Biomass

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ABSTRACT

Using water hyacinth as a phytoremediation agent produces abundant biomass due to periodic harvesting in the system. One of the alternative uses of water hyacinth biomass can be a bio-sorbent to absorb metal contamination in the waters. This study aims to determine the quality of activated water hyacinth bio-sorbent, potentially reducing metal cadmium (Cd). The research was conducted from January to April 2022. The results showed that the parameters of water content, iodine absorption, and methylene blue in water hyacinth bio-sorbent had met the quality standard of activated carbon based on SNI No. 06-3730-1995. In contrast, the ash content still did not. In water, hyacinth stem bio-sorbents (stems + $ZnCl_2$ and stems 300 °C + $ZnCl_2$) obtained higher ash content (25.87 and 73.30%) than the ash content of water hyacinth root and leaf bio-sorbent with the same activation treatment. The optimum adsorption capacity (*Qe*) for the roots + $ZnCl_2$ occurred at a contact time of 45 minutes which was 8.13 mg/g with an absorption efficiency (*Ef*) of 34.20%. For the root 300 °C + $ZnCl_2$, the optimum adsorption capacity and absorption efficiency occurred at a contact time of 8 hours, namely 9.08 mg/g and 38.66%, respectively. The optimum adsorption capacity and absorption efficiency of the leaves + $ZnCl_2$ occurred at a contact time of 4 hours, namely 7.63 mg/g and 32.12%, respectively. Meanwhile, at the leaves 300 °C + $ZnCl_2$, the optimum adsorption capacity and absorption efficiency occurred at a contact time of 8 hours with a value of *Qe* = 11.84 mg/g and *Ef* = 49.35%.

Keywords: heavy metals, cadmium, phytoremediation, water hyacinths, bio-sorbent.

INTRODUCTION

Industrial activities that are developing at this time have affected environmental conditions, where liquid waste from industry which is discharged directly into the waters, has caused very worrying pollution. Pollution due to industrial activities can cause huge losses because, generally, the industrial waste contains heavy metals, including mercury (Hg), cadmium (Cd), lead (Pb), and copper (Cu). These heavy metals are often used in the industry's production process as raw materials and main ingredients. Heavy metals dissolved in water are very dangerous for the life of organisms that live in it because they cannot degrade this type of pollutant in the environment. Heavy metals have toxic properties at specific concentrations. Heavy metals are bioaccumulating in living things (Usman et al., 2015). Cadmium metal is one of the toxic heavy metals found in wastewater as a by-product of industrial processes. Sources of cadmium waste come from metal ore processing industries, pesticides, mining, metal plating, paint stripping, textiles, and batteries (Igwe & Abia, 2006; Módenes et al., 2011; Emilia et al., 2013; Istarani & Pandebesie, 2014). Cadmium poisoning can be acute or chronic. The effects of poisoning that can be caused are lung, liver, high blood pressure, disorders of the kidney system and digestive glands, and cause bone fragility (Zumani et al., 2015).

Industrial activities that are developing at this time have affected environmental conditions, where liquid waste from industry which is discharged directly into the waters, has caused very worrying pollution. Various methods have been used to minimize Cd metal content in wastewater through physical, chemical, and biological treatments. One alternative to wastewater treatment that is environmentally friendly and economical is through a plant-based biological approach (phytoremediation). Phytoremediation uses plants and their parts for waste decontamination and environmental pollution problems. The use of phytoremediation methods can be applied both ex-situ and in-situ in areas contaminated with waste (Stefhany et al., 2013).

One of the plants commonly used in phytoremediation is water hyacinth. This type of plant can be used to overcome pollution caused by industry and households (Setyanto & Warningsih, 2011). Water hyacinth has the ability as a biofilter and has a significant sorption and accumulation capacity of specific pollutants (Setyowati et al., 2019). Water hyacinth plant can be used as an effective adsorbent in reducing heavy metals, such as Cu, Pb, Ni, Au, Co, Sr, and Sn (Rakhmania et al., 2017; Mayasari et al., 2017; Sangkota et al., 2017). The growth rate of water hyacinth ranges from 400 to 700 tons of biomass per ha per day. This plant spreads and reproduces through the formation of stolons (Tellez et al., 2008; Rakhmania et al., 2017). However, the presence of water hyacinths in the waters also has a negative impact in the form of increasing evapotranspiration, disrupting water traffic, increasing habitat for disease vectors in humans, and reducing the aesthetic value of the aquatic environment.

The use of water hyacinth as a phytoremediation agent can cause new problems because of its abundance of biomass products due to its high growth rate. For this reason, harvesting periodically and finding a solution to reuse the biomass from this water hyacinth is necessary. One alternative to post-harvesting of water hyacinth biomass is to use it as a bio-sorbent that can be reused to absorb metal contamination in the waters. Water hyacinth's bio-sorbent capacity to adsorb heavy metals can be increased through physical and chemical activation processes. Physical activation is the process of breaking carbon chains from organic compounds with the help of heat, steam, and CO_2. While chemical activation is the process of breaking the carbon chain from organic compounds by using chemicals, chemical activators that can be used include $ZnCl_2$, Na_2CO_3 NaOH, H_2SO_4, HCl, HNO_3, H_3PO_4 and others (Akhmad et al., 2012). This study aims to determine the quality of bio-sorbents from activated water hyacinth roots, stems, and leaves which have the highest potential in reducing metal cadmium (Cd).

MATERIALS AND METHODS

The bio-sorbent used in this study came from harvesting the phytoremediation process for textile wastewater carried out at the Research Centre for Limnology and Water Resources, BRIN – Indonesia. Water hyacinth is separated from the roots, stems, and leaves and cut into ± 2 cm. All biomass parts were washed with tap water, dried in the sun in a greenhouse for seven days, then dried in the oven at 65 °C for 48 hours. The dried water hyacinth biomass was mashed using a blender to facilitate filtering. Furthermore, the biomass of roots, stems, and leaves was filtered through a 200-micron sieve.

Root, stem, and leaf biomass after being sifted, weighed 50 grams, and put into an Erlenmeyer, then activated with 500 mL of $ZnCl_2$ 10% solution. The water hyacinth biomass solution was stirred for 10 minutes with a magnetic stirrer and allowed to stand for 24 hours. Furthermore, the biomass was filtered using Whatman filter paper No. 1, washed with demineralized water to neutral pH (pH 7±0.05) then heated in an oven at 105 °C for 1 hour. Bio-sorbent 1 is ready to use. The second production of bio-sorbents is by heating the biomass of water hyacinth roots and stems. It leaves at a temperature of 300 °C for 2.5 hours using a kiln, then cooling to room temperature. Furthermore, the workings of making bio-sorbent 2 are the same as the steps for making bio-sorbent one as described above. The bio-sorbent quality parameters tested in this study refer to SNI 06-3730-1995.

The preliminary test determined the part of water hyacinth biomass (roots, stems, leaves) that had the highest adsorption capacity in reducing cadmium metal (Cd). The test stage is by weighing each bio-sorbent 1 and 2 (roots, stems, and leaves of water hyacinth) of 0.5 grams. Each bio-sorbent is then put into an Erlenmeyer, then 250 mL of a standard solution of Cd 50 mg/L is added. The pH value was adjusted with 0.1 M HCl or 0.1 M NaOH solution to pH 5 ± 0.05. Then the solution was stirred with a shaker at a speed of 120 rpm for 60 minutes and filtered with 0.45 m cellulose nitrate filter paper. AAS measured the concentration of Cd ions in the solution.

The test of the effect of contact time on the adsorption capacity of the selected bio-sorbent was the result of the first preliminary test. Each selected bio-sorbent was weighed at 1 gram, then 250 mL of 50 mg/L Cd standard solution was added. The pH value was adjusted with 0.1 M HCl or 0.1 M NaOH solution to pH 5 ± 0.05. Then the solution was stirred with a shaker at a speed of 120 rpm, a contact time of 0; 0.25; 0.5; 0.75; 4, and 8 hours. The solution was filtered with 0.45 mm cellulose nitrate filter paper, then the concentration of Cd ions in the solution was measured by AAS.

RESULT AND DISCUSSION

Bio-sorbent quality

The water hyacinth bio-sorbent produced after the physical and chemical activation process is shown in Figure 1. Bio-sorbents (roots, stems, and leaves) that were preheated at T = 300 °C before being activated with $ZnCl_2$ 10% solution looked darker, and the amount of bio-sorbent produced was less than the bio-sorbent activated with $ZnCl_2$ 10% solution. According to Akhmad et al. (2012), the higher the carbonization temperature, the more activated carbon produced will be darker in color and have a greater absorption capacity. The water content, tar, and volatiles in activated carbon decrease with increasing carbonation temperature. The carbonation process in manufacturing activated carbon is a crucial step where in this process, hydrocarbon chains such as cellulose and hemicellulose are terminated into pure carbon (Nuria et al., 2020).

Moisture content, ash content, and absorption of iodine and methylene blue were tested on the bio-sorbent to determine its quality according to SNI 06-3730-1995. The results of the bio-sorbent quality test are presented in Table 1.

The quality of the bio-sorbent has met the SNI 06-3730-1995 standard on all three parameters (moisture content, absorption of iodine, and methylene blue), except for the ash content parameter. Water content determination aims to determine the hygroscopic nature of activated carbon (Sahara et al., 2017). The water content in activated carbon will affect the internal interaction process, where water will dissolve carbon and increase the molecular weight, which can

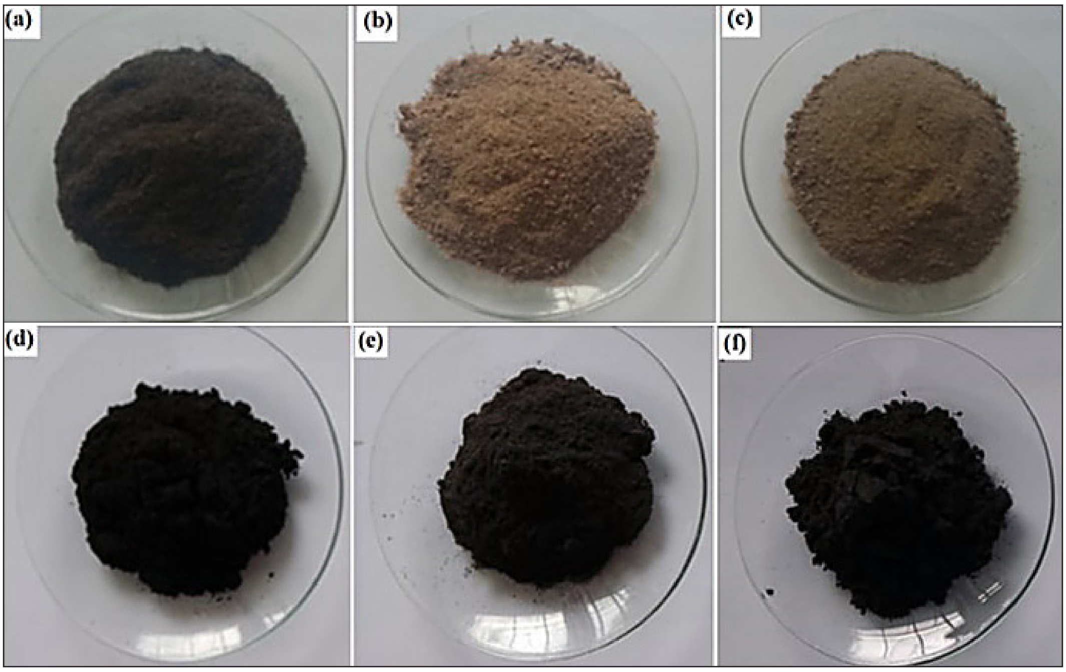

Figure 1. Water hyacinth bio-sorbent was activated by $ZnCl_2$ in (a) roots, (b) stems, (c) leaves, and activated with T = 300 °C + $ZnCl_2$ in (d) roots, (e) stems, and (f) leaves

Table 1. The quality of the water hyacinth-activated bio-sorbent on SNI No. 06-3730-1995

Parameter	SNI	Sample's Code					
		Roots $ZnCl_2$	Roots 300°C + $ZnCl_2$	Stems $ZnCl_2$	Stems 300°C + $ZnCl_2$	Leaves $ZnCl_2$	Leaves 300°C + $ZnCl_2$
Water content (%)	Max.15	8.30	3.75	10.70	2.67	9.34	4.64
Ash content (%)	Max. 10	15.30	47.44	25.87	73.30	19.55	52.64
Iodine adsorption (mg/g)	Min. 750	1281.26	1373.97	1400.63	1228.17	1267.20	1294.62
Methylene blue adsorption (mg/g)	Min. 120	199.43	196.03	1976.57	192.48	197.64	195.30

inhibit the adsorption process of activated carbon (Rahayu et al., 2022; Shah et al., 2018; Zhou et al., 2001). The water content of all types of bio-sorbents produced in this study ranged from 2.67 to 10.70%. The water content of the bio-sorbent heated at a temperature of 300 °C is smaller than that of the unheated bio-sorbent. With the increasing carbonation time and temperature, the pores of the bio-sorbent will be more open, and the water content will be released. The surface area will increase with the size of the bio-sorbent pores. This condition will increase the ability of the adsorption properties of the bio-sorbent (Sulaiman et al., 2017; Hendrawan et al., 2017).

The quality of the bio-sorbent has met the SNI 06-3730-1995 standard on all three parameters (moisture content, absorption of iodine, and methylene blue), except for the ash content parameter. Water content determination aims to determine the hygroscopic nature of activated carbon (Sahara et al., 2017). The water content in activated carbon will affect the internal interaction process, where water will dissolve carbon and increase the molecular weight, which can inhibit the adsorption process of activated carbon (Rahayu et al., 2022; Shah et al., 2018; Zhou et al., 2001). The water content of all types of bio-sorbents produced in this study ranged from 2.67 to 10.70%. The water content of the bio-sorbent heated at a temperature of 300 °C is smaller than that of the unheated bio-sorbent. With increasing carbonation time and temperature, the pores of the bio-sorbent will be more open, and the water content will be released (Sulaiman et al., 2017; Hendrawan et al., 2017).

The results of the analysis of ash content in all types of bio-sorbents are higher than SNI No. 06-3730-1995. The lowest ash content (15.30%) was found in roots + $ZnCl_2$, while the highest ash content (73.30%) was found at stems 300 °C + $ZnCl_2$. Ash content is the number of inorganic substances in the form of metals or minerals that cannot evaporate in the ashing process (Junary et al., 2015; Sulaiman et al., 2017). The high ash content in activated bio-sorbents produced in this study was due to the presence of inorganic substances in the form of metals or minerals in water hyacinth biomass which was previously used in phytoremediation for treating textile wastewater. The increase in ash content can occur because mineral salts will be formed during the combustion process, which will form fine particles of the mineral salt (Sulaiman et al., 2017). Excessive ash content will reduce the surface area of the bio-sorbent due to clogging of the pores (Junary et al., 2015).

Adsorption of iodine (iodine number) measures the number of micropores contained in activated carbon by absorbing iodine from a solution (Itodo et al., 2010; Rahayu et al., 2020). The measurement of the iodine number aims to see the adsorption ability of the bio-sorbent in absorbing the adsorbate (Sahara et al., 2017). The iodine value ranges from 1228.17 to 1400.03 mg/g, met SNI 06-3730-1995, which is a minimum of 750 mg/g. The high number of iodine produced in all types of bio-sorbents in this study indicated that all bio-sorbents had a high ability to adsorb adsorbate or solutes. According to Rahmadani & Kurniawati (2017), the parameters used to characterize activated carbon use the iodine number approach. The value of the iodine number is directly proportional to the ability of activated carbon to adsorb the adsorbate or solute.

The purpose of measuring the absorption of methylene blue (methylene blue number) is to determine the surface area of activated carbon and its ability to absorb colored solutions (Sahara et al., 2017). The value of the methylene blue number indicates the maximum amount of methylene blue dye absorbed in 1 gram of the adsorbent. The number of methylene blue ranged from 192.48 to 199.43 mg/g, which already complied with SNI No. 06-3730-1995, which is a minimum of 120 mg/g. The methylene blue number can also be used as an indicator of adsorption capacity related to activated carbon's mesoporous and macropore capacities (Rahayu et al., 2022).

Performance of activated water hyacinth bio-sorbent in reducing Cd

Preliminary tests were carried out to see the ability of each activated water hyacinth bio-sorbent in roots, stems, and leaves. The performance of these bio-sorbents in reducing Cd can be shown in Figure 2.

The adsorption capacity (*Qe*) and absorption efficiency (*Ef*) of Cd at roots 300 °C + $ZnCl_2$ were greater (11.11 mg/g and 44.47%) compared to the *Qe* and *Ef* values of roots $ZnCl_2$ (9.98 mg/g and 39,91%). The difference in the Qe value of the two bio-sorbents was 10.2%. Stems 300 °C + $ZnCl_2$ obtained a *Qe* and an *Ef* value of 5.57 mg/g and 22.28%. These results are much greater than the *Qe* and *Ef* values of the stems $ZnCl_2$ bio-sorbent. The difference in the Qe value between the bio-sorbent stems 300 °C + $ZnCl_2$ and stems $ZnCl_2$ was 76.4%.

Based on the data above, it can be concluded that the bio-sorbent, carbonized first at 300 °C before being activated with $ZnCl_2$ 10% solution, had a greater adsorption capacity. According to Akhmad et al. (2012), the carbonization temperature is directly proportional to the value of the iodine number. The higher the carbonization temperature of activated carbon, the higher the iodine value. The significant value of the iodine number can be used to describe the surface area and microporosity of activated carbon with good precision (Srisa-ard, 2014).

Different results occurred in the leaf bio-sorbent, where the value of adsorption capacity and absorption efficiency of the leaves 300 °C + $ZnCl_2$ was smaller than that of the leaves $ZnCl_2$. The difference in *Qe* and *Ef* between the two bio-sorbents above is 84.7%. This value may be due to the high ash content in the leaves at 300 °C + $ZnCl_2$ (52.64%) compared to the ash content in the leaves $ZnCl_2$ (19.55%). According to Junary et al. (2015), excessive ash content will reduce the surface area of activated carbon due to clogging of the activated carbon pores.

Based on the preliminary test, it can be concluded that the next testing stage will use four types of bio-sorbents, namely roots and leaves activated by a combination of heating 300 °C + $ZnCl_2$ 10% solution and root and leaf bio-sorbent activated by $ZnCl_2$ 10% solution.

Effect of contact time on adsorption capacity

The bio-sorbent used in this test was selected from the previous preliminary test, root dan leaves activated with 300 °C + $ZnCl_2$. The test results data are presented in Table 2. The optimum adsorption capacity of the roots $ZnCl_2$ occurred at a contact time of 0.75 hours (45 minutes) which was 8.13 mg/g with an absorption efficiency of 34.20%. Meanwhile, for roots 300 °C + $ZnCl_2$, the optimum adsorption capacity and absorption efficiency occurred at a contact time of 8 hours, namely 9.08 mg/g and 38.66%, respectively. The same thing also happened to leaves $ZnCl_2$, where

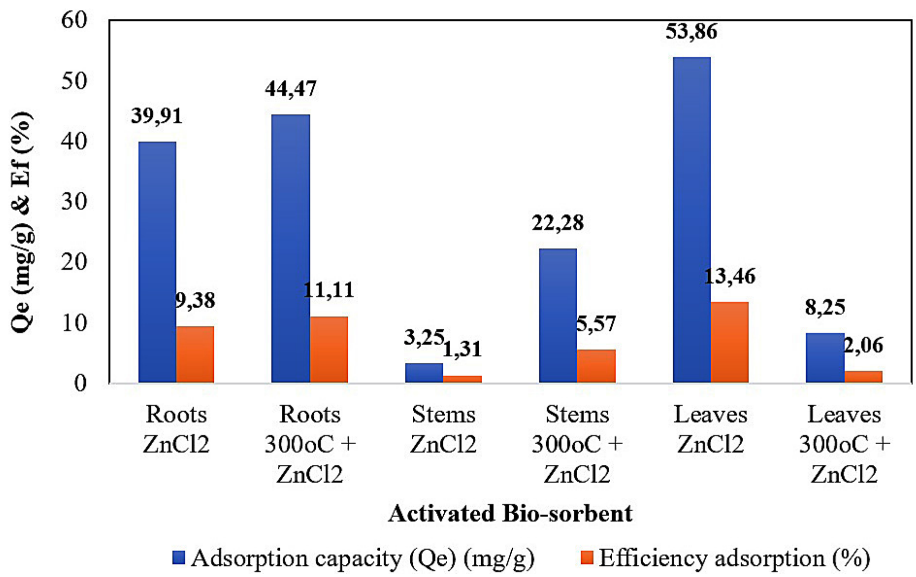

Figure 2. Adsorption capacity (*Qe*) and absorption efficiency (*Ef*) of Cd by activated water hyacinth bio-sorbent (roots, stems, leaves)

Table 2. *Qe* and *Ef* activated bio-sorbent on Cd(II) (pH 5, weight 2 g/L) with variations in contact time

Time contact (hour)	Roots $ZnCl_2$		Roots 300°C + $ZnCl_2$		Leaves $ZnCl_2$		Leaves 300°C + $ZnCl_2$	
	Qe (mg/g)	*Ef* (%)	*Qe* (mg/g)	*Ef* (%)	*Qe* (mg/g)	*Ef* (%)	*Qe* (mg/g)	*Ef* (%)
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.25	5.05	21.26	1.22	8.02	6.17	25.97	3.97	16.71
0.50	6.80	28.62	3.23	13.59	7.29	30.66	5.37	22.56
0.75	8.13	34.20	5.04	18.42	7.15	30.11	6.53	27.44
4.00	4.99	21.44	5.85	24.62	7.63	32.12	8.62	40.10
8.00	7.38	31.16	9.08	38.66	7.41	31.17	11.84	49.35

the adsorption capacity and optimum absorption efficiency occurred 4 hours faster than leaves 300 °C + $ZnCl_2$, at a contact time of 4 hours with 7.63 mg/g and 32.12%. At leaves 300 °C + $ZnCl_2$, the optimum adsorption capacity and absorption efficiency occurred at a contact time of 8 hours with a value of *Qe* = 11.84 mg/g and *Ef* = 49.35%.

From the data above, it can be concluded that the optimum adsorption capacity of Cd on bio-sorbent, which was directly activated with $ZnCl_2$, occurred faster than the bio-sorbent, which was carbonized first. The active site on the surface of the bio-sorbent activated by $ZnCl_2$ was faster in the adsorption of Cd metal to reach its equilibrium. According to Estiaty (2012), adsorption equilibrium is a condition where the rate of particle adsorbed to the surface is equal to the desorption rate.

In roots $ZnCl_2$ and Leaves $ZnCl_2$, the adsorption capacity value increased rapidly at the beginning of the contact time and tends to decrease with increasing contact time. This condition shows that there has been an equilibrium followed by the saturation of the bio-sorbent's surface so that it can no longer adsorb metal ions (Mayasari et al., 2017). The opposite happened to the root and leaves 300 °C + $ZnCl_2$, where Cd absorption required a long contact time to reach its optimum adsorption capacity. In these two bio-sorbents, the value of adsorption capacity and absorption efficiency tended to increase with increasing contact time. This phenomenon may be related to the higher ash content of roots and leaves 300 °C + $ZnCl_2$ bio-sorbent compared to the ash content of roots and leaves $ZnCl_2$ bio-sorbent (Table 2). As previously explained, the high ash content will reduce the surface area of the activated carbon, which results in clogging of the pores on the activated carbon, so that it can inhibit the Cd adsorption process rapidly by the roots and leaves bio-sorbent 300 °C + $ZnCl_2$.

Table 2 shows no significant difference between the adsorption capacity of the bio-sorbents derived from roots $ZnCl_2$and roots 300 °C + $ZnCl_2$. The difference between the two bio-sorbents was only 10.49%. Meanwhile, there was

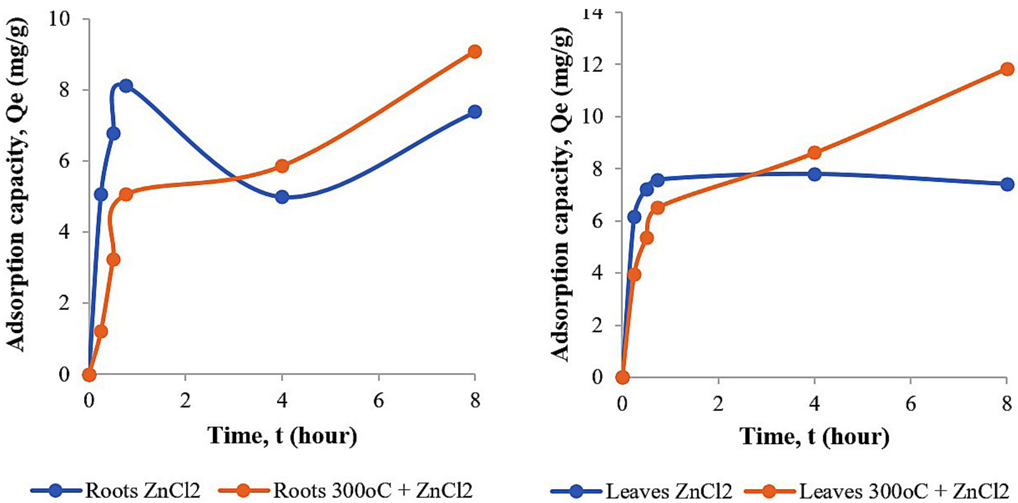

Figure 3. (a) adsorption capacity of root-activated bio-sorbent, (b) adsorption capacity of leaves-activated bio-sorbent

a significant difference for bio-sorbents derived from leaves of $ZnCl_2$ and leaves of 300 °C + $ZnCl_2$, which is the *Qe* of leaves 300 °C + $ZnCl_2$ was 35.55% higher than that of leaves $ZnCl_2$. Comparing the adsorption capacity of the roots $ZnCl_2$ to the leaves, $ZnCl_2$ obtained a higher *Qe* of 6.08%. Meanwhile, there is a reasonably high difference between the *Qe* value of the root and leaves 300 °C + $ZnCl_2$, where the *Qe* value in the leaves is 23.34% higher than the roots.

CONCLUSION

The bio-sorbent, which was activated by heating and chemically (T = 300 °C and $ZnCl_2$ 10%), visually looks darker with less weight than the bio-sorbent with chemical activation ($ZnCl_2$). The quality of the bio-sorbent on the parameters of ash content, iodine, and methylene blue adsorption, has met the quality standard of activated carbon based on SNI No. 06-3730-1995. However, the ash content still does not meet the standard. The ash content of water hyacinth stems with two thermal and chemical activation treatments was higher than roots and leaves with the same activation treatment. Bio-sorbent with two activation treatments has a higher adsorption capacity (*Qe*) than bio-sorbent with chemical activation only. Roots and leaves bio-sorbents with chemical activation of $ZnCl_2$, the *Qe* on Cd increased rapidly at the beginning of the contact time. They tended to decrease with increasing contact time. Bio-sorbent with two activation treatments needs a longer contact time to reach its optimum adsorption capacity. In these two bio-sorbents, *Qe* and *Ef* on Cd tend to increase with increasing contact time. Significant differences were found in activated leaves with $ZnCl_2$ and activated leaves with 300 °C and $ZnCl_2$ up to 35.55%. The best bio-sorbent that gives the best performance to the uptake of Cd was by using the leaves of water hyacinth dried biomass which was activated by thermal in 300 °C and chemical with $ZnCl_2$ 10%.

Acknowledgments

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