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Biosorption Performance of Biochar from Exhausted Kahwa Coffee on Cadmium Removal Under Various Operational Parameters

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ABSTRACT

The primary goal of this study was to investigate the biosorption performance of biochar derived from exhausted kahwa coffee (EKC) on removing heavy metal ion, Cd^{2+} in the solution. The operational parameters, for instance pH, dosage and contact time, were varied in a certain range and the efficiency and the capacity of removal were determined. The pH was in a range of 5 – 11, while the biochar dosage in a range of 0.075 – 0.15 g and the contact time was in between 30 – 180 minutes. The batch biosorption test showed that the EKC biochar had higher removal performance in alkaline condition, with a proposed pH of 10. Meanwhile, the applied dosage showed that addition of 0.1 g EKC biochar was more effective than adding more dosage. Furthermore, the batch experiments showed that 60 minutes of biosorption process resulted in more effective removal compared to the longer biosorption time. According to the ANOVA analysis on the three conditions, it was shown that the pH and contact time had significant effects on the removal performance of EKC biochar on cadmium ion in the solution.

1. INTRODUCTION

The rapid industrialization has led to severe heavy metal pollution in aquatic ecosystems, posing significant ecological risks (Mitra *et al*., 2022; Saini & Dhania, 2020). Among these heavy metals, cadmium is particularly toxic and is classified as a class 1 carcinogen within agroecosystems (Danapriatna *et al*., 2024; Zhao *et al*., 2023). Exposure to cadmium on agricultural lands can profoundly impair plant growth by disrupting various physiological and metabolic processes. These adverse effects include mitochondrial dysfunction, chlorosis, necrosis of plant tissue cells, and inhibition of root and shoot development, ultimately diminishing the nutritional quality of plants (Hayat *et al*., 2018; Sasidharan & Kumar, 2022). Consequently, it is imperative to regulate cadmium levels in the environment to mitigate its detrimental impacts, especially within the agricultural sector.

The employment of biosorbents derived from agro-industrial waste is recognized as an efficient and economical approach for mitigating the presence of heavy metal ions, including cadmium ions (Cd^{2+}), in aquatic environments (Chen *et al*., 2019; Li *et al*., 2017; Rusnam *et al*., 2024). Numerous agro-industrial solid wastes have been assessed for their efficacy as biosorbents for Cd^{2+}, including wheat bran (Singh *et al*., 2006), rice husk (Sobhanardakani *et al*., 2013), coffee husk (Oliveira & Franca, 2015), sawdust (Long *et al*., 2021), and exhausted kahwa coffee (EKC) (Yanti *et al*., 2022). Despite the extensive generation of coffee waste in the food industry, the use of EKC biochar remains relatively unexplored. EKC biochar exhibits promising potential for cadmium ion removal under various biosorption activation processes, while also promoting the principles of a circular economy within the agricultural sector (Puari *et* *al*., 2024; Yanti *et al*., 2022). However, there is a paucity of research investigating the biosorption performance of EKC under different carbonization parameters.

The efficiency of biosorbents in removing heavy metals is influenced by various factors, including the type of biomass, the specific heavy metal ion, the biosorbent preparation, and environmental parameters during the biosorption process (Torres, 2020). Understanding the interplay among these factors is essential for predicting the performance of a given biosorbent and achieving optimal heavy metal reduction in wastewater. Key environmental parameters, such as pH, biosorption time, and biosorbent dosage, significantly impact the chemical processes during biosorption and the subsequent removal outcomes. Different precursor of biochar and the heavy metal ion targeted would result in specific key parameters (Faisal *et al*., 2024; Sasidharan & Kumar, 2022). Therefore, this study focuses on investigating the effects of operational parameters on the biosorption of cadmium ions using EKC biochar. Analysis of variance (ANOVA) will be utilized to assess these effects and identify the most critical factors.

2. METHODOLOGY

2.1. Materials

Exhausted Kahwa Coffee (EKC) for the experimental studies were obtained from the local kahwa coffee shop in Tanah Datar region, West Sumatera, Indonesia. The standard solution of cadmium ion (Cd^{2+}) was prepared by dissolving 1000 mg/L of $Cd(NO_3)_2 \cdot 4H_2O$ with ultrapure water. Sodium hydroxide solution (NaOH) (0.5 N) was used to adjust the solution pH into the desired values. All chemicals used in the experimental studies were analytical grade and purchased from local supplier of Merck in Indonesia.

2.2. Experimental Procedure

• Biochar preparation

Samples of EKC as raw material were washed and then dried using an oven (MEMMERT UN 55) at 105°C for 4 hours. The preparation of EKC Biochar production was in accordance with previous research by Yanti *et al*. (2022). Dried EKC was underwent carbonization using muffle furnace at temperature of 500°C for 120 minutes (Nabertherm B180). Each carbonization process took 20 grams of the dried EKC and placed in a porcelain cup covered with aluminum foil at temperature. Eventually, the biochar was crushed using a mortar and pestle, then sifted using a 50-mesh sieve to get the desired particle sizes.

• pH adjustment

Shortly after adding the biochar into the standard solution in the Erlenmeyer flask, the pH was adjusted using NaOH 0.5 N solution into the desired values. The pH value was measured with pH meter (FiveEasy Plus pH meter FP20, Mettler Toledo).

• Biosorption

Biosorption process was carried out under batch method by placing the Erlenmeyer flask with the solution on a digital orbital shaker (SK-0330 PRO Nesco Official). The 100 mL of solution with the addition of biochar was shaken under 150 rpm at room temperature. Biosorption experiments were conducted in seventeen combinations of operational parameters (pH, dose of biochar and contact time between the biochar and Cd^{2+} metal), as the details were presented in Table 1. Each experimental set were conducted in triplicate condition, and standard deviation were determined to evaluate the data range. While one parameter was varied, the other two parameters were kept constant.

After biosorption process, the mixture solution was filtered using filter paper (Whatman 42) to separate the solution and EKC biochar. The concentration of the cadmium ion in the filtered solutions were then measured using Atomic Absorption Spectrometry (AAS) (Shimadzu AA-6880) analysis. To determine the concentration of Cd^{2+} in the solution, absorption values of Cd^{2+} standard solutions were measured and plotted against concentration to obtain a calibration graph as presented in Figure 1.

Table 1. Biosorption experimental set with the various combinations of operational parameters

Parameter	pH	Dosage (g)	Contact time (min)
pH	5	0.1	60
	6		
	7		
	8		
	9		
	10		
	11		
Dosage	optimum pH	0.075	60
		0.1	
		0.125	
		0.15	
Impregnation	optimum pH	Optimum dosage	30
			60
			90
			120
			150
			180

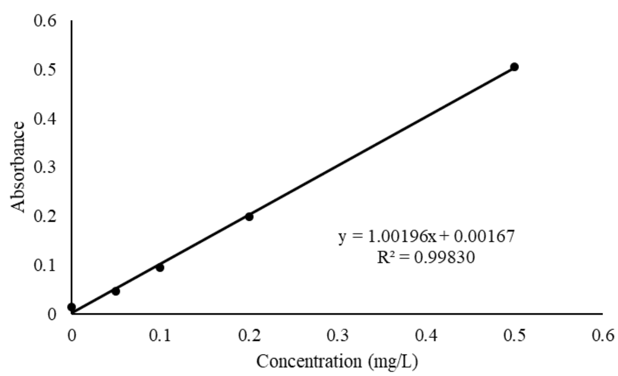

Figure 1. Calibration curve of Cd^{2+} at different concentration.

The percentage removal efficiency (RE) (Eq. 1) and the biosorption capacity (qt) (Eq. 2) of the EKC biochar on Cd^{2+} removal, were calculated by the following equations:

$$\boldsymbol{RE}\ (\%) = \frac{C_o - C_t}{C_o}\ x\ 100\% \quad (1)$$

$$q_t\ (\mathbf{mg/g}) = \frac{(C_o - C_t)\ V}{m} \quad (2)$$

where, C_0 is the initial concentration of Cd^{2+} (mg/L), C_t is final concentration of Cd^{2+} after biosorption, V is the volume of the solution (mL), and m is dose of the biochar used (g) (Zhou *et al*., 2019).

2.3. Data Analysis

A one-way analysis of variance (ANOVA) was utilized to determine the significance of variations among the biosorption experiments with EKC biochar under different operational parameters. The analysis was conducted at a 99% confidence level, with a probability threshold set at $p < 0.01$. Statistical evaluations were carried out using SPSS Statistic 22.0 software. To ensure the assumptions of the ANOVA test were met, all data underwent a normality test beforehand. Moreover, the mean values from triplicate biosorption experiments were used for graph plotting.

3. RESULTS AND DISCUSSION

3.1. pH Value

To explore the effect of pH value on biosorption of Cadmium, the experiment was carried out under various pH values, which were between 5 until 11. The initial pH of the mixing biochar and the solution is 2. The pH solution was adjusted by adding NaOH 0.1 M. Figure 2 exhibited the removal efficiency (RE) and biosorption capacity (q_t) of EKC biochar on Cd^{2+} removal within the pH range, with a contact time of 60 minutes and dosage of 0.1 g. It was clearly seen in the graph that varied solution pH resulted in different removal outcomes. The efficiency and the capacity of EKC biochar were seen increased when the solution in more alkaline condition. The lowest RE and qt from the experiments was seen when the solution pH was at 5 with 22.71 ± 7.84% and 5.87 ± 2.12 mg/g, respectively. The contrast results were depicted when the pH of the solution increased to 11, with RE was 99.56 ± 0.26% and q_t was 25.70 ± 0.59 mg/g.

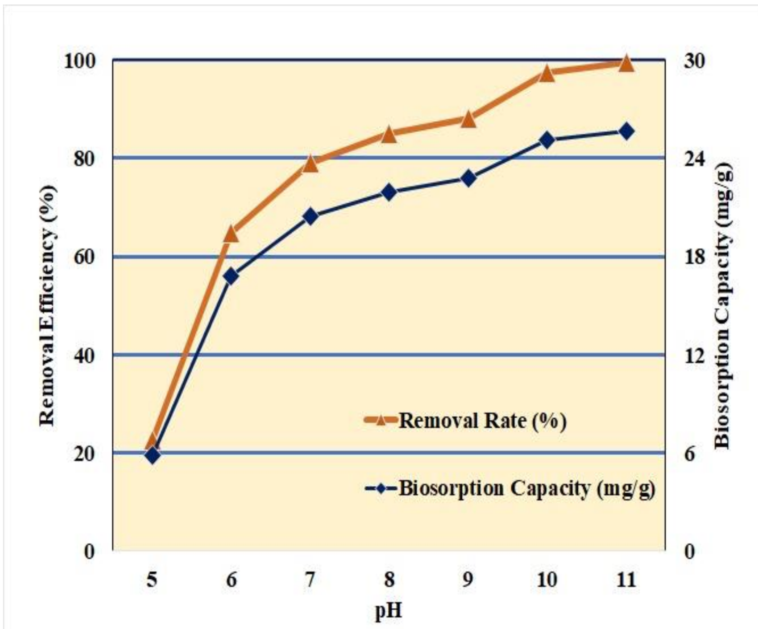

Figure 2. Effect of pH value on Cd removal at constant dose of 0.1 g and contact time of 60 minutes.

The experimental result were in line with previous research conducted by Anisuzzaman *et al.* (2015) and Wang *et al.* (2022). Both studies showed removal of Cd ion with other biomass increased with the increasing pH, as the pH solution is related to competition between ions during biosorption. It was stated in the previous study that in certain pH, ions in solution can compete with target ions that will be absorbed by the biosorbent. Furthermore, it is also related to the metal ability to form hydroxide or carbonate complexes at a certain pH. In this study, the RE was increased by the increasing pH, which led the assumption of the removal of Cd ion related to the presence of hydroxide ion. Changes in solution pH can affect the surface charge of biochar, thereby providing an opportunity for heavy metals to coordinate with functional groups which can increase removal efficiency (Paranavithana *et al.*, 2016). It has been supported by the findings in the previous studies which showed that the functional groups of biochar play roles in the removal heavy metal ion by decreasing of its intensity in the infrared observation (Liu *et al.*, 2019; Puari *et al.*, 2024). Moreover, high removal in alkaline region might also be affected by the precipitation process of the metal ion aside of the biosorption by the EKC biochar, as for Cd the precipitation happens at pH ≥ 8 (Wang *et al.*, 2022). Experimental results showed that the pH of the solution can influence the absorption efficiency and biosorption capacity, where a higher pH can increase the removal efficiency of cadmium ion.

Meanwhile, it is also interesting to discuss the final pH of the solution after underwent biosorption process as presented in Table 2. The data showed that the final pH solutions were always neutral with a pH range from 6.18 to 6.54 when the biosorption started when pH solution were at pH 5 to 10. The neutral solution after the biosorption process might be due to the ion presence has been removed during the biosorption. However, an exception result was noticed when the biosorption conducted at the initial pH solution of 11. At the certain pH, the final pH solution after biosorption process was highly in alkaline condition, which was 10.42 ± 0.11. Even though the RE reached 99.56 ± 0.26% with a qt of 25.70 ± 0.59 mg/g at pH 11, however, it was unable to change the pH to neutral that occurred during absorption. There is no research that specifically states that the higher the pH can increase the efficiency and capacity of metal absorption, this goes back to the nature of the biochar and the type of metal in the solution being experimented.

Table 2. Initial pH and final pH of the solution during biosorption process

Initial	5.0	6.0	7.0	8.0	9.0	10.0	11.0
Final	6.18 ± 0.13	6.55 ± 0.03	6.67 ± 0.01	6.48 ± 0.05	6.66 ± 0.02	6.54 ± 0.28	10.42 ± 0.11

It is noteworthy to state that determination of the best pH for the absorption of certain metals is crucial for predicting the performance of a given biosorbent and so that the pH is optimal for metal absorption. In this study, the one-way ANOVA test showed that there was a significant relationship ($p < 0.01$) between solution pH and the Cd^{2+} removal percentage of the EKC-BC. It is well understood from the test results that the solution pH determines the biosorption capability of the biochar. Hence, pH 10 was chosen as the optimum condition in this study, where RE was obtained 97.35 ± 0.65% and qt 25.12 ± 0.39 mg/g, and the final pH obtained was already in a neutral pH state of 6.54 ± 0.28.

3.2. Dosage

The investigation on the effect of biosorbent dosage on biosorption process of EKC biochar was done by varying the biochar dosage from 0.075 g to 0.15 g while the initial pH solution and the biosorption time were constant. The effect of various biochar dosage on the elimination of Cd ion in term of RE and qt of EKC biochar was illustrated in Figure 3. It was clearly seen from the figure that experimental results showed a positive trend on RE, while for the q_t had a negative one. The results showed that the percentage of metal ion removal increased along with increasing the amount of adsorbent used. While the highest RE achieved when the highest dosage added to the solution with 98.3 ± 0.58%, on the contrary it resulted in the lowest qt with 16.13 ± 0.58 mg/g.

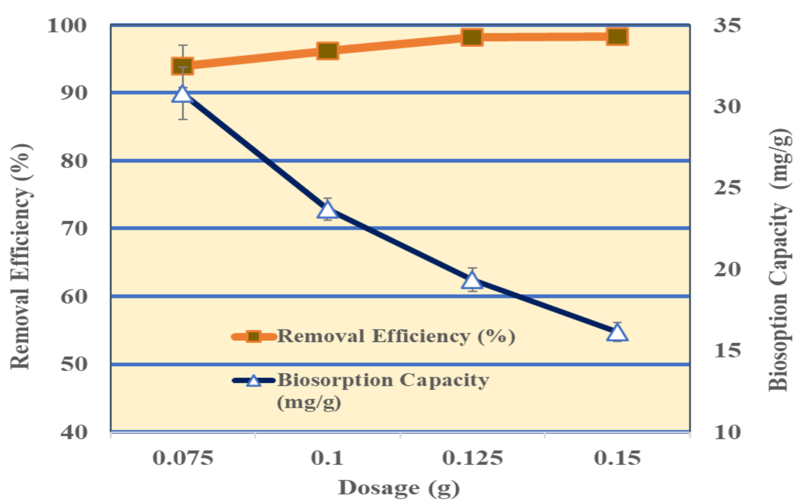

Figure 3. Effect of biochar dose on Cd removal at constant pH Value 10, and contact time 60 minutes.

The findings from these experimental studies showed the same as the findings in previous research by Azizi *et al*. (2012) that the absorption capacity of Cd metal decreased as the dose concentration increased, because biomass aggregation occurred at high doses (higher than 1.6 mg per 100 mL solution), reducing the absorption surface area so that the effectiveness of the biosorption area was reduced. Meanwhile, Ali & Bhakta (2020) also reported that at certain doses, adsorbent particles collect at a higher concentration so that they can block absorption. In this study, the dosages applied were in a range of 75 – 150 mg. Furthermore, it was discussed by Puari *et al*. (2024) and Ahmadi *et al*. (2022) in their previously published studies, that the cause of the trends were due to the increase in the specific surface area and pore volume available for biosorption with increasing adsorbent dosage. Interestingly, the statistical analysis using the one-way ANOVA test on the RE from the experimental results showed that there was no significant relationship ($p > 0.01$) between the dosage of biochar and the percentage of the EKC-BC removal efficiency on Cd (II). However, the ANOVA test between on the qt and dosage of biochar showed a significant relationship ($p < 0.01$). It can be concluded that adding the excessive dosage of the biochar do not give a significance on the RE outcomes. However, the significance is notable in term of the qt of the biochar as it has a dependable not only on the amount of the targeted pollutant removed but also on the amount of added biosorbent to the solution. According to the obtained results and ANOVA analysis, therefore dosage of 0.1g of EKC biochar was determined as the suggested dosage for the Cd removal. Even though the RE could be achieved higher by adding higher biochar, however increasing was only less than 2%.

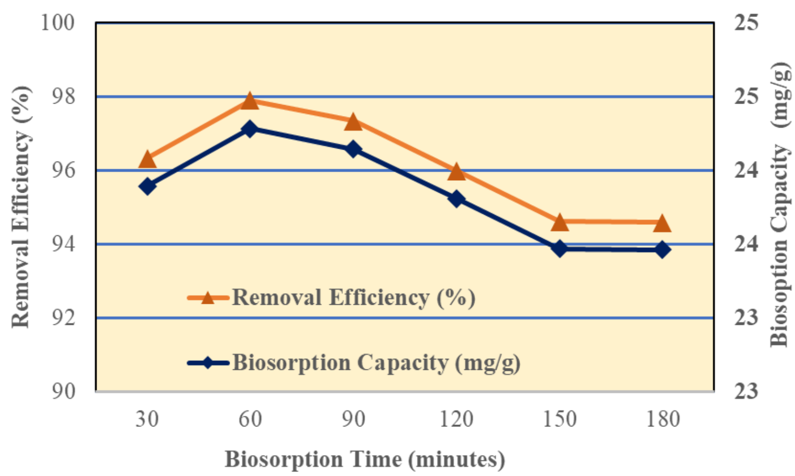

Figure 4. Effect of biosorption time on Cd removal at constant pH value 10 and dose of biochar 0.1 g.

3.3. Biosorption Time

In all sets of experiments, fixed volume of metal solution in 100 mL at pH 10 was stirred with the 0.1 g of biochar dosage for the period of 30 min to 180 min. Figure 5 displayed the effect trend of biosorption time on the RE and qt of EKC biochar. It was clearly seen in the figure that the EKC biochar could remove almost 94% of Cd ion in the solution at each variation of time. The highest removal of Cd achieved when the biosorption run for 60 minutes with RE 97.89 ± 0.39% and qt 24.3 ± 0.16 mg/g. Initially, there was an increase in RE and qt when extending the biosorption time from 30 to 60 minutes. However, when the extension time continued to 90 minutes decreasing trend was noticed and eventually it continued until the biosorption time of 180 minutes with 94.59 ± 0.67% of RE and 23.46 ± 0.14 mg/g of qt. The similar results were also reported in the previous study by Faisal *et al*. (2024), where the study also had an increasing of Cd ion removal percentage with different biomass in the first 30 minutes of adsorption. The study exposed that after 90 minutes the removal rate was no longer significant due to the faster depletion of the absorption sites.

Phuengphai *et al*. (2021) stated in their study that contact time of each biosorbent depends on the equilibrium point. Furthermore, it was also explained that fast biosorption rate in the beginning due to a larger surface area biosorbents available for metal biosorbent. The decreasing trend after 60 minutes could be due to the biosorbent reached saturated point, which is resulted in some absorbed heavy metal ions were leached back to the solution. Meanwhile, the statistical analysis using the one-way ANOVA test on the biosorption data showed that there was a significant relationship ($p < 0.01$) between contact time and the percentage of the EKC-BC removal efficiency and the biosorption capacity on Cd (II). Based on experiments, 60 minutes was determined as the suitable biosorption time for absorbing Cd ions.

4. CONCLUSION

Exhausted Kahwa Coffee Biochar (EKC BC) was used as a biosorbent to investigate the removal of Cd ions from simulated wastewater in batch experiments. The research concluded that operational parameters, namely pH, dosage of biochar and contact time had significant effect on the performance of EKC biochar for Cd ion removal in the solution. Basic condition at pH in between 10 and 11 is more suitable for Cd ion removal by EKC biochar, with 60 minutes of contact time. The percentage of removal efficiency of EKC biochar on Cd (II) at basic condition in this study reached 99.56%. The addition of dosage biochar from the experimental results was 0.1 g with the capacity of biosorption was 23.68 mg/g.

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