

Characterization of Biosorbent from *Musa acuminata balbisian* Peel using FTIR Spectroscopy and Its Application to Cadmium (Cd) Removal: Effect of Activator Type, pH, and Biosorbent Ratio

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ABSTRACT

*Biosorbent that use in this study made from kepok banana peel (*Musa acuminata balbisian*). The present study is done to determine the characteristic of biosorbent and the effect of activator type, pH, and ratio of biosorbent for the cadmium removal. The experiments were carried out in batch process, laboratorium scale, room temperature, and 60 mesh of biosorbent size particle. The biosorbent produced by using chemical activation method onto dry banana peel. The variation in this research was the type of activator (H_3PO_4 and H_2SO_4), pH of Cadmium (3, 4, and 5), and the ratio of biosorbent dosage to the volume of cadmium solution (1:20, 1.5:20, and 2:20 (g/ml)). The biosorbent characterized by using Fourier Transform Infra-Red (FTIR). The cadmium concentration analyzed by using Atomic Absorption Spectroscopy (AAS). Characterization with FTIR showed the differences in functional groups onto biosorbent before and after adsorption. There was a change in the spectral band of C-H and C=O functional groups for biosorbent that activated with H_3PO_4 and a change in the spectral band of C-H functional groups for biosorbent that activated with H_2SO_4 . The acid activators showed the best condition was at 90 minutes contact time, pH 4 and the ratio of biosorbent dosage and the volume of 1.5:20 (g/ml). The result showed that the H_3PO_4 was the best acid activator in the removal of cadmium with amount of 99.91%.*

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1. Introduction

Cadmium metal is widely used in the metal alloy industry, metal coating, electronics, and textiles. Cadmium discharged into water will cause damage to the body's physiological systems such as breathing, blood circulation, smell and vital organs [1]. Therefore, grade II cadmium carrying heavy metal content according to Government Regulation No. 82 of 2001 concerning Water Quality Management and Water Pollution Control should not exceed 0.01 mg/l [2].

One of the alternatives in managing waste containing heavy metal is by using biological materials as biosorbent with a process called biosorption. Among the advantages of using biosorption process are the relatively low cost, high efficiency, and ease of the regeneration process. Various alternative agricultural waste products can be used as a biosorbent raw material such as rice husk, beet powder, and banana peel [3] [4] [5].

Kepok banana (*Musa acuminata balbisan*) is one type of tropical fruit with abundant availability with production reaching 7.162 million tons based on the release of the Indonesian Central Statistics Agency in 2017 [6]. The amount of banana peels is about 1/3 of the unpeeled bananas [7]. Banana peels contain lignin (6-12%), pectin (10-21%), cellulose (7.6-9.6%) and hemicellulose (6.4-9.4%) [8]. Pectin compounds in banana peels can be used to remove excess cations or heavy metals [9].

Percentage of the absorption of cadmium metal towards activated carbon from the banana peel is 98.35%, 89.65%, and 64.2% [3] [9] [10]. On the other hand, the cadmium metal biosorption with banana peel biosorbent has the percentage of 89.2%, and 73.15% [11] [12]. Likewise, the percentage of the absorption from the durian skin is 72.17% and 64.54% [12] [13]. Referring to the average percentage of cadmium metal adsorption between biosorbent and activated carbon that is not too significant. The activated carbon is obtained from pyrolysis method and/or chemical activation, while the biosorbent is obtained from chemical activation. Production of biosorbent is simpler than activated carbon. This experiment does the absorbing process of cadmium metal using *kepok* banana peel as the biosorbent. The characteristic of biosorbent is analyzed by using Fourier Transform Infra-Red (FTIR) method. The effectivity *kepok* banana peel as biosorbent is tested to adsorb cadmium metal in aqueous solution by modifying the activator type. The effect of pH and biosorbent ratio is also determined in the adsorption process.

2. Research Methodology

2.1. Materials

Materials used in the research were *kepok* banana peel (*Musa acuminata balbisan*), aquadest, H₃PO₄ (technical solution, 6 M), H₂SO₄ (technical solution, 2 M), Cd (cadmium) solution (Synthetic solution, 2 ppm). The tools used were Atomic Absorption Spectroscopy (AAS), Fourier Transform Infra-Red (FTIR), erlenmeyer 25 ml, measuring cup 25 ml, beaker 500 ml, volumetric flask 250 ml, stirring rod, spatula, oven, magnetic stirrer, filtering paper, funnel, blender, 60 mesh sieve, pH meter, analytical balance, and sample bottle.

2.2. Procedures

1) *The Making of Kepok Banana Peel Biosorbent*

The inside of *kepok* banana peel (*Musa acuminata balbisan*) is cleaned and then washed with water. The clean banana peel is dried under the sun for approximately 48 hours. The size of dry banana peel is reduced to 60 mesh, next will be called as biosorbent.

2) *Activating biosorbent using H₃PO₄ and H₂SO₄ as an Activator*

The activator used in this research was H₃PO₄ and H₂SO₄. The activator and biosorbent are mixed with ratio 2:1 by using a magnetic stirrer at the speed of 90 rpm for 30 minutes. After that, the biosorbent is rinsed by using aquadest until neutral or the pH near 7. Then the neutralized biosorbent is dried by using an oven at a temperature of 105 °C for 2 hours.

3) *Application of adsorption process*

The cadmium solution is made according to the Indonesian National Standard (SNI) 6989.16:2009 with pH 3, 4, and 5. Then, 1 gram, 1.5 gram and 2 gram of biosorbent is mixed with 20 ml cadmium solution and stirred with the speed of 90 rpm for about 10, 45, 90 and 120 minutes. This procedure is repeated for another variation. The experiments were carried out in batch process, laboratory scale, and room temperature [28]. The solution is filtered and then the filtrate is analyzed by using Atomic Absorption Spectroscopy (AAS) [29]. The fresh and reused biosorbent is characterized by using Fourier Transform Infra-Red (FTIR).

2.3. Data Analysis

The ability of biosorbent in adsorb of the cadmium is called Adsorption Efficiency (% Removal) that can be calculated with the equation (1), where C_o and C_e are the initial and final concentration of Cd (mg/l) [28].

$$\% \text{Removal} = \frac{C_o - C_e}{C_o} \times 100\% \quad (1)$$

3. Results and Discussion

3.1. Characteristics of Biosorbent made from *Kepok* Banana Peel (*Musa acuminata balbisian*)

Functional group analysis method in a sample based on absorbing spectra infra-red through FTIR [14]. Infra-red spectrum can be obtained through a light transmission that passes the sample and light intensity measuring with a detector. Infra-red spectrum that has been obtained then is plotted as heat function intensity and wavelength (cm^{-1}) [15]. FTIR spectrum on *kepok* banana peel before and after being in contact with cadmium solution is approximately $4000\text{-}500\text{ cm}^{-1}$ to identify functional groups in biosorbent.

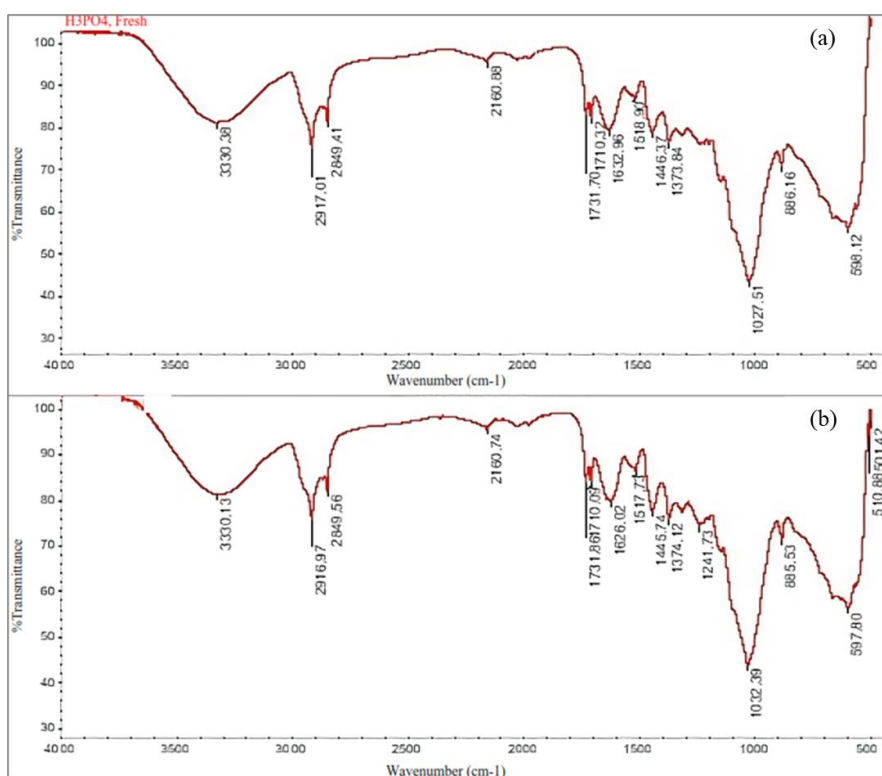


Fig. 1. FTIR spectrum of *kepok* banana peel biosorbent with activator H_3PO_4 (a) fresh, (b) reused

The FTIR spectra of durian and banana peel materials were before and after cadmium ion biosorption. These spectra were obtained from scanning in the range of $400\text{-}4000\text{ cm}^{-1}$. The band of durian and banana peel represented overlapping of O-H and N-H stretching vibration at 3369 and 3388 cm^{-1} , respectively. The band represented C=O stretching vibration of carboxylic acids at 1741 and 1731 cm^{-1} , respectively. The significant shifts of these specific peaks to the higher wave number after the cadmium ions biosorption suggested that chemical interactions between the cadmium ions and the amide groups occurred on the biomass surface. The influences of the shift peaks were very similar for the fruits peel materials. The band of durian, and banana peel shifted to 3416 and 3416 cm^{-1} . The spectra of banana and durian peel at 1741 and 1731 cm^{-1} , respectively became smoother and a band of banana peel at 1427 cm^{-1} , respectively, appeared, which would result from the complications of cadmium ions with the functional groups from protein. These results indicated that carboxyl, hydroxyl and amide groups on the fruit peels' surfaces were involved in the biosorption of the cadmium ions [12].

FTIR spectrum *kepok* banana peel biosorbent with activator H_3PO_4 before and after being in contact with Cd solution can be seen on the Fig. 1. Fig. 1(a) shows that both H_3PO_4 activated biosorbents have hydroxyl O-H peak whose wavelength vibration is 3300 cm^{-1} that explains that there is a hydroxyl group free of a polymeric compound like lignin or pectin that has functional group like alcohol, phenol, and carboxylic acid. This matches the approximated frequency for

hydroxyl group between $3600\text{--}2800\text{ cm}^{-1}$ [16]. Also, the result of the characterizing shows the existence of C-H and C=O group with a wavelength of 2917 cm^{-1} and 1626 cm^{-1} .

Fig. 1 (b) shows that the absorption band formed at wave number 1241 cm^{-1} which is the absorption of the amine C-N functional group. In both spectrums, it can be seen that the biosorbent of *kepok* banana peel that has been contacted will experience a decrease in transmittance value and a change in absorption band with a shift in wavelength from 2917 cm^{-1} to 2916 cm^{-1} for the C-H functional group. Meanwhile, in the functional group C=O there is a shift in the absorption band which indicates the vibration and the transmittance value decreased from 1632 cm^{-1} to 1626 cm^{-1} . Shifting the absorption band that occurs in each functional group in the biosorbent and the appearance of a new peak indicates that the cadmium metal is absorbed on the surface of the banana skin biosorbent. Based on the identification of the functional groups from the FTIR analysis it was proven that the biosorbent of the *kepok* banana peel was activated H_3PO_4 effective in absorbing cadmium metal.

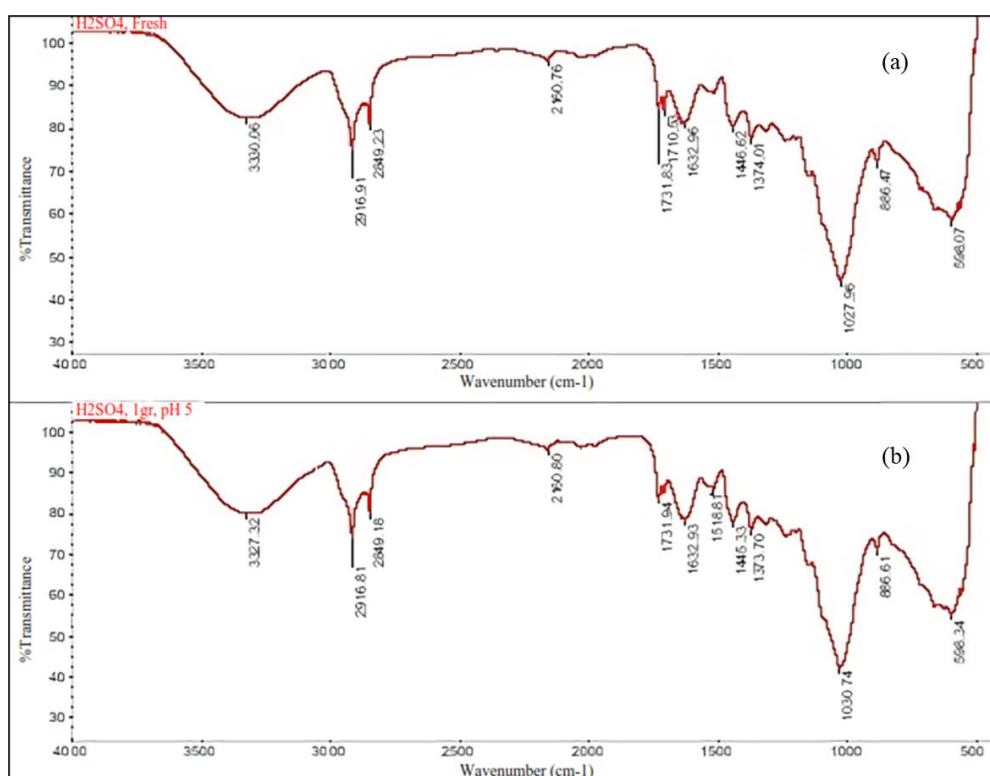


Fig. 2. FTIR Spectrum of *kepok* banana peel biosorbent with activator H_2SO_4 (a) fresh, (b) reused

FTIR spectrum of *kepok* banana peel biosorbent with H_2SO_4 activator before and after contacting is seen in the Fig. 2. Fig. 2 (a) proves the existence of O-H functional groups that are characterized by wavelengths ranging from $3330\text{--}3327\text{ cm}^{-1}$. The next absorption band appears at wave number 2916 cm^{-1} which is the absorption of the aliphatic and aromatic C-H functional groups. Wavenumber 1632 cm^{-1} appears as a third absorption band with a wavelength of $1675\text{--}1500\text{ cm}^{-1}$ indicating the presence of C=O functional groups which can be acidic compounds, aldehydes, ketones, amides, esters, anhydrides, in this case, the bonds are perhaps included in the third absorption area is the C=O ketone functional group because in the lignin structure there are only C=O ketone bonds. However, in Fig. 2 (b) a new absorption band is formed at the wave number 1518 cm^{-1} which is the absorption of the N-O functional group.

Fig. 2 (b) shows that the biosorbent of *kepok* banana peel activated H_2SO_4 after contacting did not change the transmittance value and absorption band for the functional groups C-H and C O. This shows that cadmium metal only binds to O-H functional groups and N-O functional groups as new peaks. The results of the characterization of banana skin biosorbents prove that H_2SO_4 -activated *kepok* banana peel biosorbents are not very effective when used as biosorbents to absorb cadmium

metal. The comparison of functional group presented in the biosorbents based on different activator type is shown in Table 1.

Table 1. The Results of FTIR Analysys

Activator	Frequency Range (cm ⁻¹)	Functional Group
H ₃ PO ₄	3300	Hydroxyl Compound
	1241	Amine C-N
H ₂ SO ₄	2916	The aliphatic and aromatic C-H
	1518	N-O Nitro Compound

3.2. The Effect of Activator Type, pH and Biosorbent Ratio to Removal of Cadmium

pH is a parameter in maximizing the adsorption capacity of solution metal ions. In this case, pH affects the surface properties of the adsorbent and the ionic shape of the cadmium solution. Cadmium adsorption is sensitive to changes in acidity or alkalinity conditions so that proper pH is needed in cadmium adsorption [17]. [18] in his research, proved that at pH below 2, the percentage of cadmium metal absorption is very low. However, at high pH, i.e above pH 7, the percentage of absorption of cadmium metal is also not optimum due to the precipitation of cadmium to Cd(OH)₂ in the solid phase. Therefore, the absorption process of cadmium metal in this study occurred at pH 3, 4 and 5.

Fig. 3 shows a graph of the percentage of cadmium adsorption at pH 3 with a weak acid activator H₃PO₄. Cadmium absorption is good, indicated by the percentage of adsorption in the range of 65.35-95.87%. Similar to the H₂SO₄ strong acid activator, Fig. 3 shows good cadmium absorption with adsorption percentage of 61.41-97.64%. However, the absorption of cadmium metal at pH 3 in both H₂SO₄ and H₃PO₄ activators is still unstable. At low pH, high concentrations of hydrogen ions cause fewer surface functional groups available in the biosorbent to bind cadmium metal. The best condition at pH 3 with H₃PO₄ weak acid activator was at 45 minutes and the adsorbent dose was 1.5 grams while the best condition at pH 3 with strong acid activator H₂SO₄ was shown at 120 minutes and the adsorbent dose was 2 grams.

As known, H₂SO₄ is a strong acid that has higher ionization energy compared to H₃PO₄ [19]. High ionization energy at higher H₂SO₄ activators produces a more acidic atmosphere and causes more H⁺ ions to be present on the surface. This causes more competition between H⁺ and Cd²⁺ ions so that the absorption of cadmium metal at pH 3 requires a longer time and more adsorbent dose to get the best absorption conditions. In contrast, the ionization energy of H₃PO₄ is not so high that it does not require a lot of time and a dose of adsorbent to achieve the best conditions in the absorption of cadmium metal. However, acidic conditions that are too concentrated at pH 3 cause the cadmium ions attached to the biosorbent are unstable and easily detached from the surface. [20] also states that effective desorption of heavy metals is carried out at low pH.

The graph of the percentage absorption of cadmium metal at pH 4 with H₃PO₄ and H₂SO₄ activators respectively is shown in Fig. 4. The absorption of cadmium at pH 4 is more stable when compared to pH 3. Under acidity conditions at pH 4, it also produces the best average percent absorption with a range of percentage absorption at 97.3-99.91% for H₃PO₄ and 96.5-98.8 % for H₂SO₄. The best conditions for the absorption of cadmium metal in this study with H₃PO₄ and H₂SO₄ activators were located at 90 minutes and 2 grams of adsorbent dose. This is because at pH 4 the concentration of hydrogen ions in the biosorbent is less so that it provides more functional groups on the surface of the biosorbent to densely absorb cadmium metal.

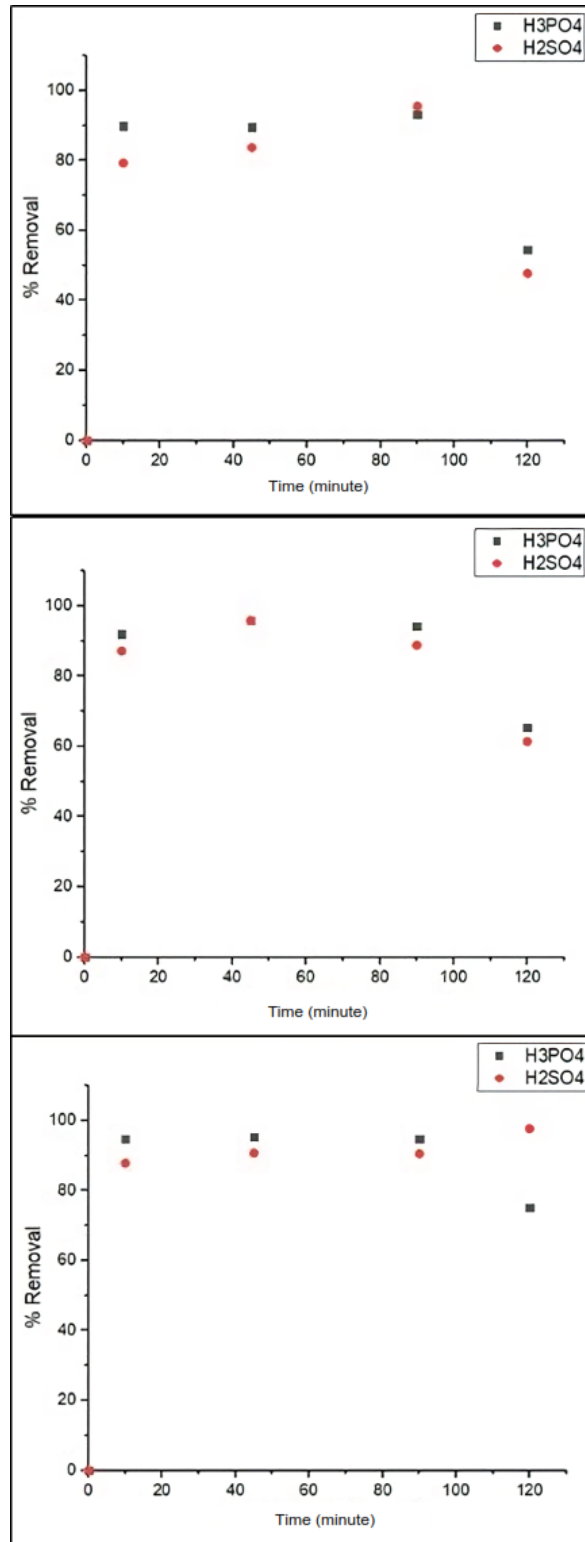


Fig. 3. The Effect of absorbent dosage towards cadmium metal adsorption using activator H₃PO₄ and H₂SO₄ on pH 3 with dosage (a) 1 gr ; (b) 1.5 gr ; (c) 2 gr

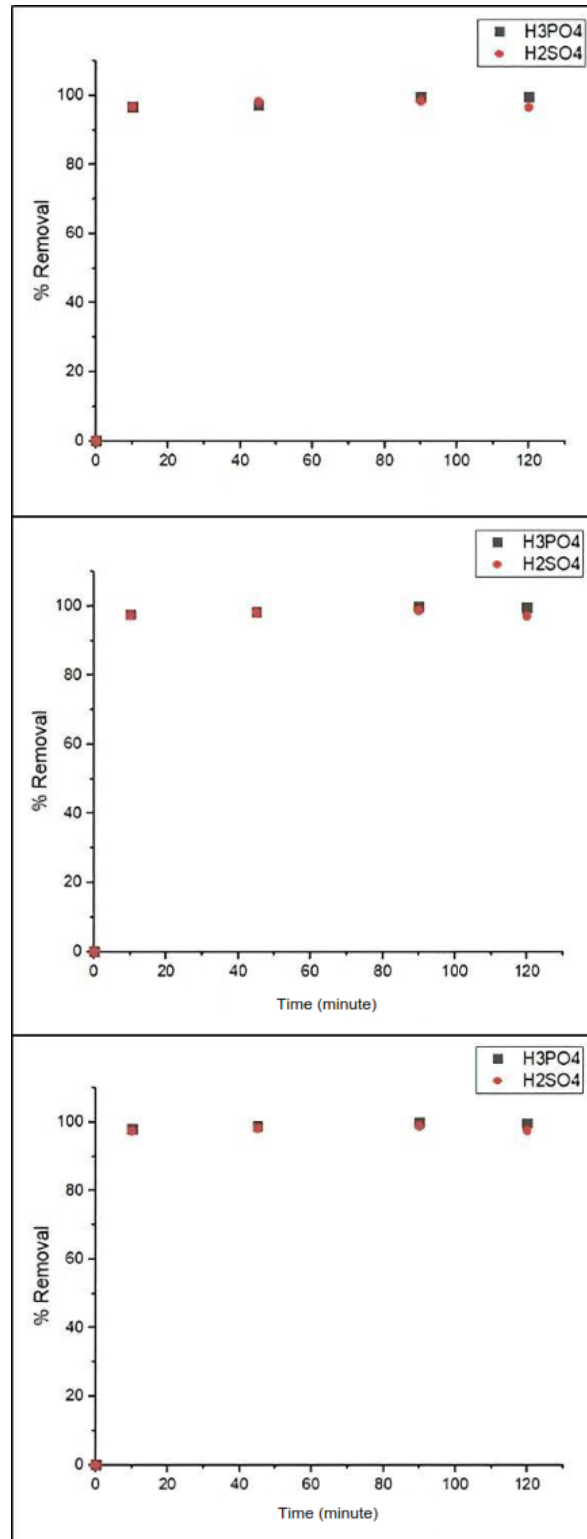


Fig. 4. The Effect of adsorbent dosage towards cadmium metal adsorption using activator H₃PO₄ and H₂SO₄ on pH 4 with dosage (a) 1 gr, (b) 1,5 gr, (c) 2 gr

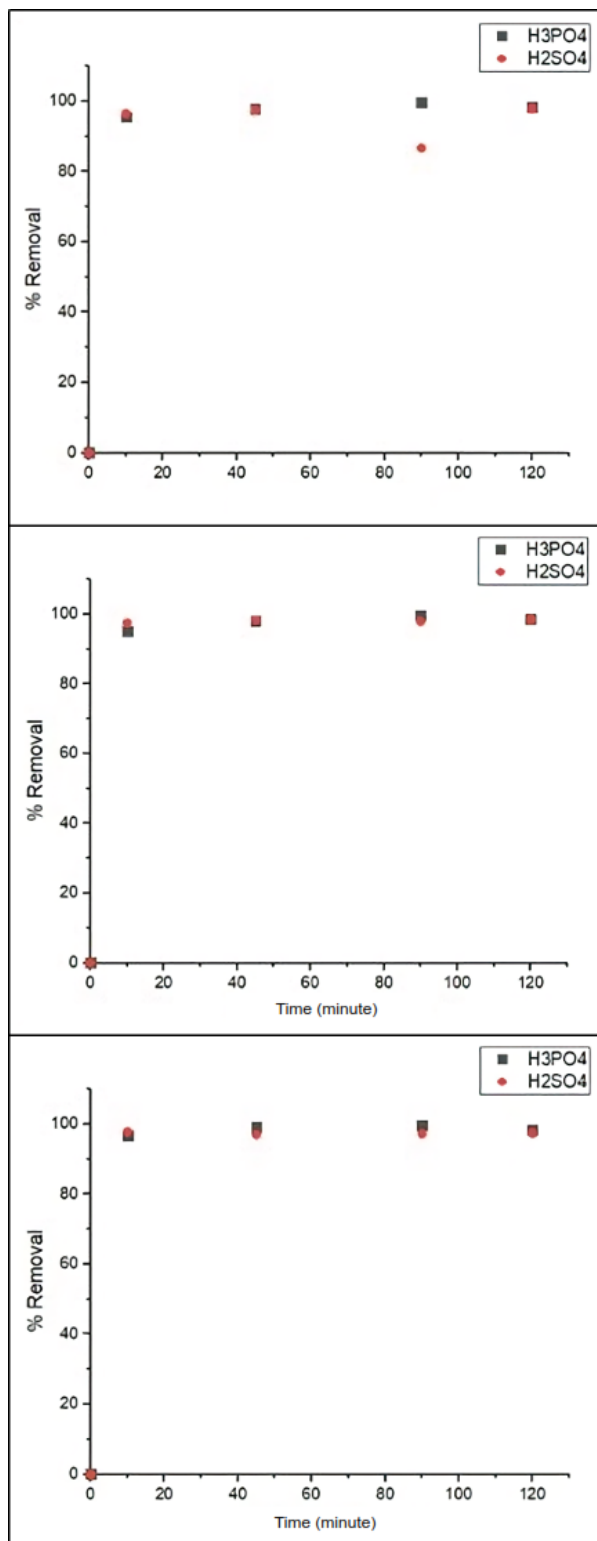


Fig. 5. The Effect of adsorbent dosage towards cadmium metal adsorption using activator H₃PO₄ and H₂SO₄ on pH 5 with dosage (a) 1 gr, (b) 1.5 gr, (c) 2 gr

The absorption of cadmium metal at pH 5 with H₃PO₄ and H₂SO₄ activators is approximately the same as pH 5 with an adsorption percentage range of 95.5-99.76% for H₃PO₄ activators and 86.68-98.5% for H₂SO₄ activators. The data is presented in Fig. 5. The best conditions for the absorption of cadmium metal with an H₃PO₄ activator at pH 5 occurred in the 90th minute with a

dose of 2 grams of adsorbent. In contrast, the best conditions for absorption of cadmium metal with H₂SO₄ activator occurred in the minute 90 with a dose of 1.5 grams of adsorbent.

In essence, metal absorption increases with increasing doses of the adsorbent. However, cadmium concentrations under certain conditions in H₃PO₄ and H₂SO₄ activators have increased after the adsorption process, although not significantly. The addition of biosorbent doses under certain conditions may cause aggregation of biosorbents. Aggregation can reduce the biosorbent density, causing the absorption of cadmium metal to decrease because the active site on the biosorbent is reduced [21].

3.3. The comparison of biosorbent ability in Cadmium degradation

The best acid activator for the absorption of cadmium metal with biosorbent *kepok* banana peel in this study is the H₃PO₄ solution. [3] and [22] explained the classification of metal ions into metal cations of type A (strong acids), transition metals and type B (weak acids) according to the acidity of a solution with a tendency towards ligands and metal stability [22, 23]. Based on these classifications, cadmium is a type B metal cation that is more suited to weak acid activators, in this case, phosphoric acid. Type B metal cations have a tendency to bind to S and N ligands. This description supported the reason for the absorption band in the biosorbent of *kepok* banana peel with H₃PO₄ activator in Fig. 3 (b) where the wavenumber 1241 cm⁻¹ is formed which is the absorption of the CN amine functional group.

Phosphoric acid has a pKa value of 2.15-7.12 while the carboxyl bonds in the *kepok* banana peel pectin which are an important group in the absorption of cadmium metal have pKa of 3.5-5.0 [24]. The corresponding pKa values between the two create the right acid atmosphere so that the carboxyl bonds are deprotonated into negative ions (-COO⁻). In these circumstances, the cadmium metal adheres to the carboxyl bonds so that the percentage of cadmium absorption becomes maximum. On the other hand, sulfuric acid has a low pKa of -3 resulting in an acid atmosphere that is too thick. This causes competition between H⁺ ions and cadmium metal and causes the cadmium metal absorbed to be unstable so that the percentage of cadmium absorption goes up and down. There was some adsorbent that ever used as agent to remove cadmium in aqueous solution that showed in Table 2.

Table 2. Comparison of cadmium removal using different adsorbents

Adsorbent Type	% Removal	Activator	Reference
Active Carbon Banana Peel	98.35	-	[25]
Active Carbon <i>Kepok</i> Banana Peel	64.2	H ₂ SO ₄	[10]
Banana Peel Biosorbent	89.2	-	[11]
Banana Peel Biosorbent	85	-	[26]
Banana Peel Biosorbent	73.15	-	[12]
<i>Durian</i> Skin Biosorbent	72.17	-	[12]
Active Carbon <i>Durian</i> Skin	64.54	HCl	[13]
Orange Skin Adsorbent	93.72	HCl	[27]
<i>Kepok</i> Banana Peel Biosorbent	99.91	H ₃ PO ₄	Present Study
<i>Kepok</i> Banana Peel Biosorbent	98.81	H ₂ SO ₄	Present Study

4. Conclusion

Biosorbent that use in this study made from *kepok* banana peel (*Musa Acuminata balbisan*). The type of H₃PO₄ activator which is in the form of a weak acid produces the best percentage of absorption in the cadmium removal when it compared with the H₂SO₄ activator. Characterization with FT-IR showed the differences in functional groups onto biosorbent before and after adsorption. There was a change in the spectral band of C-H and C=O functional groups for biosorbent that activated with H₃PO₄ and a change in the spectral band of C-H functional groups for biosorbent that activated with H₂SO₄. Both of the activator types showed the best condition was at 90 minutes

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contact time, pH 4 and the ratio of biosorbent dosage and the volume of 1.5: 20 mg/l. The result showed that the H_3PO_4 was the best acid activator in the removal of cadmium with amount of 99.91%.

Notation

C_o = the initial concentration of Cadmium (Cd) (mg/l)

C_c = the final concentration of Cadmium (Cd) (mg/l)

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