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Adsorption of chromium (VI) ion using adsorbent derived from lignin extracted coir pith

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ABSTRACT

In this study, coconut coir pith (cellulose-lignin compound) was first treated with water and sodium hydroxide solution to remove lignin and impurities, then lignin-extracted coir pith was calcined at 200℃ for 6 h. The obtained adsorbent was applied to remove Cr(IV) ions by using adsorption method. Some analytical methods such as thermal gravimetric analysis (TGA), Fourier Transform Infrared Spectroscopy (FT-IR), Brunauer–Emmett–Teller analysis (BET), X-ray powder diffraction analysis (XRD), pH_{pzc} analysis, Boehm titration, and potentiometric titration were employed to characterize structure, specific surface area, functional groups, and surface charge of the adsorbent. Adsorption results showed that 95.23% of Cr(IV) was removed from solution of 100 $mg.L^{-1}$ by using a certain amount of adsorbent at pH 2.0 within 20 min at room temperature. Kinetics of Cr(VI) adsorption from aqueous solution on adsorbent fit to Pseudo-second-order kinetic equation and adsorption isotherm of Cr(IV) followed to the Freundlich model.

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1 INTRODUCTION

Among industrial wastewaters, electroplating effluent discharged is one of the most dangerous wastewaters, which normally contains high concentration of heavy metal ions such as Cr(VI), Cr(III), Cu(II), Zn(II), and Ni(II), etc. due to low yields of electroplating process (Zhao *et al.*, 1999). Those ions are non-biodegradable, toxic, and carcinogenic, leading to an extremely environmental concern on the wastewater. Compared to other ions, Cr(VI) ions have obtained plenty of interest from researchers owing to its negative effects and commercial value (Kimbrough *et al.*, 1999; Silva *et al.*, 2006; Cavaco *et al.*, 2007). From the environmental point of view, the sustainable wastewater treatment should completely recycle treated water to producing lines (Peng *et al.*, 2004). In the case of Cr(VI) ion removal, it might be realized by adsorption using carbon derived from agricultural by-products (Tinh, 2011; Do *et al.*, 2016), red mud (Dung *et al.*, 2015), and distillery sludge (Selvaraj *et al.*, 2003). Those studies reported that agricultural by-products are highly potential to remove Cr(VI) ion via their porous structure, but original adsorbents still meet their limits; hence, it is necessary to modify their surficial properties to improve the adsorption ability.

In Vietnam, coconut coir pith (CCP) is very abundant and a by-product of coconut fiber production, causing environmental pollution in terms of water. It is a cellulose-lignin compound with low carbon content, but high surface area activated carbon can be produced from CCP (Bansal and Goyal, 2005). Santhy and Selvapathy (2004) created an adsorbent with surface area of 877 $m^2.g^{-1}$ due to the fact that the CCP was sunk in KOH 10% solution and calcined at 700 – 750°C before washing with HCl 10% solution. Besides, CCP was washed and dried under sunlight for 5 h before being activated with $ZnCl_2$ at ratio of 1:2 (w/w), and then calcined at 700°C for 1 h. The product possessed high cationic exchange ability (1.614 $mEq.g^{-1}$) with specific surface area of 910 $m^2.g^{-1}$ (Namasivayam and Sangeetha, 2006). One study reported that CCP calcined at different temperatures of 400, 600, and 800°C, respectively created different specific surface areas (346, 392, and 507 $m^2.g^{-1}$) (Namasivayam and Kadirvelu, 1997). The surface areas increased with rising calcined temperature, but carbon generated yields decreased from 60 to 55.2% as temperature increased from 400 to 800°C (Namasivayam and Kadirvelu, 1997). In order to reutilize CCP, the present work was conducted to produce activated carbon. The adsorbent was then applied to remove Cr(VI) ion in aqueous solution with various affecting parameters.

2 MATERIALS AND METHODS

All chemicals used in this work such as sodium hydroxide pellets, potassium dichromate, phosphoric acid, sodium bicarbonate were analytical reagents and purchased from Xilong Scientific. CCP was bought from Ben Tre province, Vietnam. Distilled water was used.

2.1 Adsorbent preparation

CCP collected was pretreated to remove tannin and lignin by sinking into water and NaOH solution for a desired time. CCP was first sunk in water for 24 h to remove tannin, and then dropped into NaOH 5% solution for 24 h. The solid after that was washed until pH of the solution was neutral. The CCP was dried at 60°C for 48 h using Memmert SFE 600 dryer before calcining by Thermolyne 47900 furnace at 200°C for 6 h, with the ratio of CCP to H_3PO_4 of 1:4. After calcination, the solid was sunk into $NaHCO_3$ 1% solution for 12 h and then washed with distilled water several times to neutralize the surface of particles. Those particles were consequently dried at 60°C until the weight of solid was unchanged.

2.2 Characterization of the adsorbent

The adsorbent was characterized by using several analytical methods. TG 209 F3 Tarsus (NETZSCH) and ASAP 2020 (Micrometrics) were employed to conduct thermal gravimetric analysis (TGA) and specific surface area determination for the adsorbent. In the meantime, functional groups, morphology, and structure of the adsorbent were characterized by using Nicolet 6700 Spectrometer (Thermo Scientific), JSM 7600F (JEOL), and D8 Advance (Brucker), respectively. Boehm titration and potentiometric titration were used to determine the quantity of functional groups and the amount of charge on carbon surface.

2.3 Adsorption experiments

The desired amount of adsorbent was poured into a 250-mL Erlenmeyer flask containing 100 mL of solution with various concentrations of Cr(VI) ion. The pH values of solution varied from 1.0 to 8.0 while temperature of solution changed from 25 to 60°C. A certain amount of solution was sampled at different time intervals. The concentration of Cr(VI) ion in samples was determined by using Jenway's 6800 Spectrophotometer (Keison). All experiments were triplicated. The removal yield H(%) was calculated by the following equation:

$$\mathrm{H}(\%) = \left[\frac{\mathrm{C_0} - \mathrm{C_t}}{\mathrm{C_0}}\right] \times 100\% \qquad (1)$$

Where H, C_0 and C_t were the adsorption yield (%), initial concentration of Cr(VI) ion ($mg.L^{-1}$) and concentration of Cr(VI) ion at sampling time ($mg.L^{-1}$), respectively.

In this study, the adsorption model was built by using Langmuir and Freundlich isotherm models as follows:

Langmuir isotherm model:

$$\frac{\mathrm{C_e}}{\mathrm{q_e}} = \frac{\mathrm{C_e}}{\mathrm{q_{max}}} + \frac{1}{\mathrm{q_{max}K_L}} \qquad (2)$$

Freundlich isotherm model:

$$\log \mathrm{q_e} = \log \mathrm{K_F} + \left(\frac{1}{\mathrm{n}}\right) \log \mathrm{C_e} \qquad (3)$$

where q_e, K_F, n and C_e were mass of adsorbed ions at the equilibrium ($mg.g^{-1}$), the Freundlich constant, the index number of Freundlich equation, and concentration of solution at the equilibrium ($mg.L^{-1}$), respectively, q_{max} is described the maximum mass of adsorbed ions ($mg.g^{-1}$), K_L is the Langmuir constant.

3 RESULTS AND DISCUSSION

3.1 Adsorbent preparation

Table 1 showed that 20 g of CCP generated 10.93 g, 10.56 g, and 10.03 g of adsorbents in triplicated experiments. Consequently, the calcination yield was about 52.54% in this study. In comparison with the study of Namasivayam and Sureshkumar (1997), the carbonization yield in this work is lower by 3.0-7.0%. The reasons could be that the yield was calculated based on dried weight of CCP in this work and CCP was also pretreated before carbonizing process. In the meantime, the previous study did not conduct a pretreatment experiment and calculated the carbonization yield by a ratio of carbon to dried raw material. In addition, the authors carbonized CCP for 1 h at higher temperatures (400-800°C) without removing tiny particles after calcination so it could cause a slightly higher yield compared to this work.

Table 1: The yield of adsorbent preparation at 200°C for 6 h with CCP:H_3PO_4 = 1:4 (w/v)

	1st trial	2nd trial	3rd trial
Dried CCP (g)	20	20	20
Dried adsorbent (g)	10.93	10.56	10.03
Yield (%)	54.66	52.80	50.16
Average yield (%)	52.54 ± 2.26		

3.1 Adsorbent characterization

The adsorbent was analyzed by TGA to understand weight change, and the result was presented in Figure 1. It can be seen that when temperature increased from 30°C to 100°C, the weight of adsorbent decreased by 11.17%. If the temperature continuously increased to 250°C, a decrease of the weight was 6.43%. The total of 17.6% decrease in weight can be considered as moisture of adsorbent, consisting of 11.17% of water physical bonding and 6.43% of water chemical bonding. Figure 1 showed that in the range of 250-600°C, the weight of adsorbent decreased by 28.8% due to degradation of hydrocarbon compounds into CO and CO_2 gases. The cause of this decrease was contributed to non-complete carbonization of CCP, which occurred at relatively low temperature (200°C) for 2 h.

XRD result of adsorbent in Figure 2 showed a low intensity peak at $2\theta = 25°-26°$, which represented an amorphous carbon phase. Similar conclusions could be observed in reports of Das *et al.* (2000) and Tongpoothorn *et al.* (2011). Besides, no other peaks could be found in the XRD pattern, so it can be concluded that phosphoric acid or other phosphate compounds were totally removed by washing right after calcination.

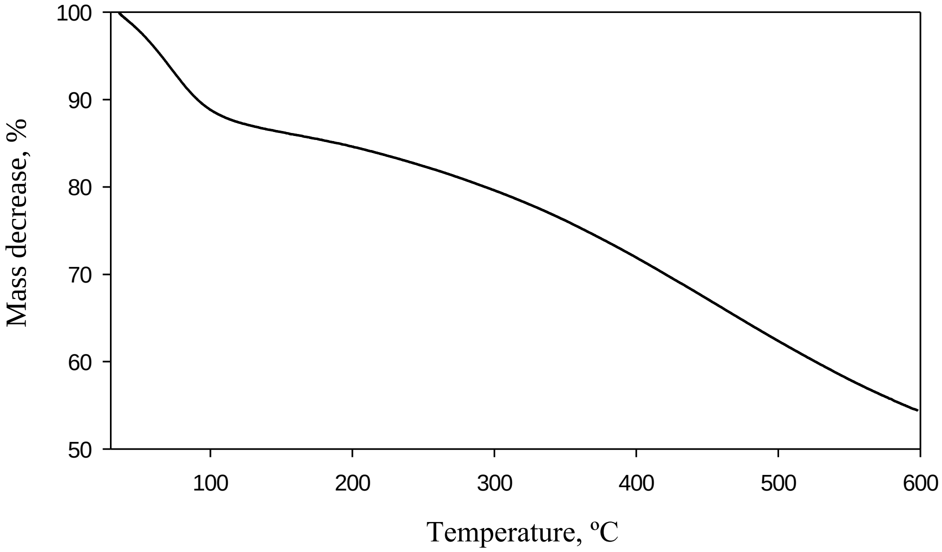


Figure 1: TGA curve of the adsorbent

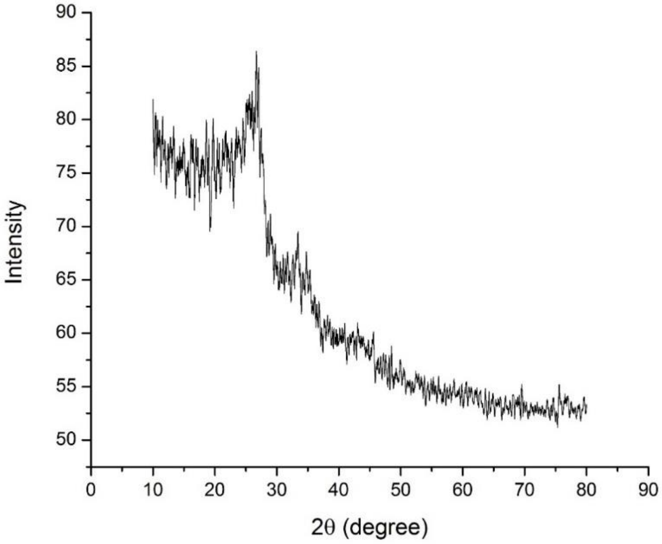


Figure 2: XRD pattern of the adsorbent

Phosphoric acid was employed in this study to carbonize CPP; thus, it could play an important role in the carbonization process. Phosphoric acid can be an acidified agent and/or functionalized agent. This was clarified by FT-IR spectra. The spectra in Figure 3 showed that peak at 3415.82 cm^{-1} was a broadband of -OH group, belonging to hydrogen-bonded alcohols or phenols. Stretching vibration of C-H (aromatic rings) and vibration of C=O group (carboxylic acids or aldehydes) were bands located at 2923.57 cm^{-1} and 1617.40 cm^{-1}, respectively. The C–O stretching vibration in carboxylate groups caused the band at 1385.45 cm^{-1}. Finally, the O–H out-of-plane bending vibration caused the band located at 565.20 cm^{-1}. Similar results can be found in the previous works of Bansal *et al*. (2009) and Yang and Qiu (2010). From all the above-mentioned groups, some positions on surface of the adsorbent were functionalized to acidic groups. In other words, carbons on the edge of adsorbent were acidified to functional groups to improve their adsorption ability, especially counter-charged solutes. No phosphate groups could be observed in FT-IR spectra of the adsorbent. It was once again concluded that phosphoric acid only played a role of acidified agent and supported carbonization agent, which could not create any bonding between phosphorus and surface of the adsorbent.

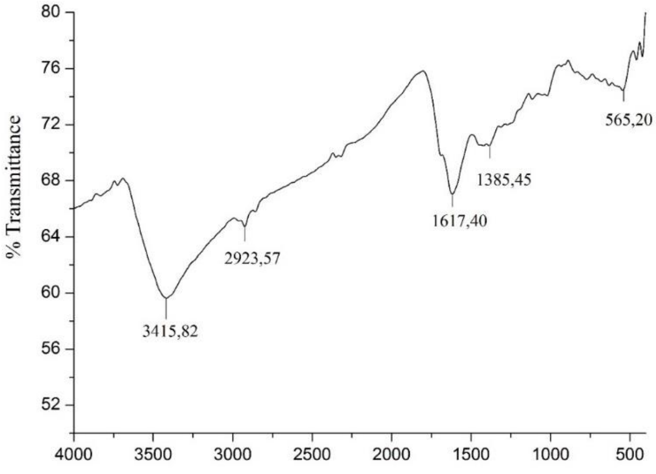


Figure 3: FTIR spectra of the adsorbent

Result of specific surface area measurement was presented in Figure 4. It can be seen that 48.56 $m^2.g^{-1}$ is a measured specific surface area of the adsorbent and this value is relatively low compared to 877 $m^2.g^{-1}$ (Santhy and Selvapathy 2004) calcining at 700-750°C in presence of KOH 10% solution or 346 $m^2.g^{-1}$, 392 $m^2.g^{-1}$, and 507 $m^2.g^{-1}$ (Namasivayam and Kadirvelu, 1997) calcining at 400°C, 600°C, and 800°C, respectively. It could be stated that temperature greatly affects specific surface area of adsorbent obtained from calcination of CCP. The higher calcining temperature applied, the stronger carbonization of CCP happened, leading to damage of internal structure of CCP to generate more pores and improve surface area of adsorbent. In addition to surface area determination, diameter of the pores was an essential factor for adsorption treatment and adsorption selectivity of adsorbent. In this study, the average pore size of the adsorbent was 10.2 nm. This could be considered as meso-pores, so several tens square meters per gram surface area of the adsorbent measured was reasonable.

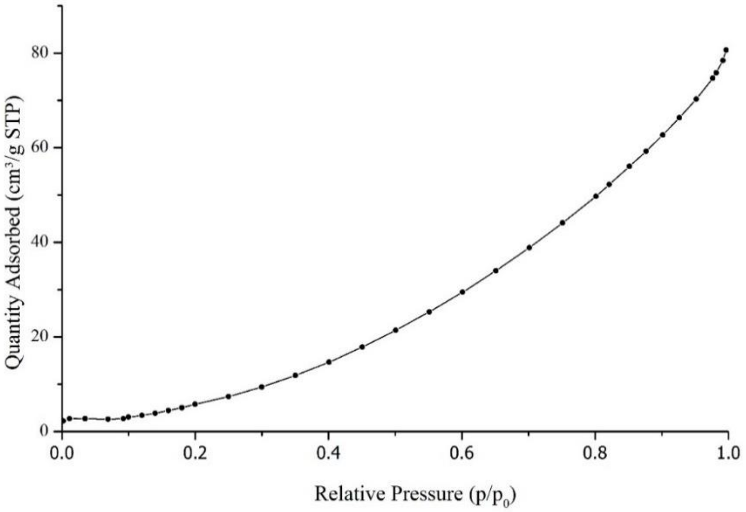


Figure 4: BET measurement of the adsorbent

Determination of acidic points on surface of adsorbent plays an essential role in prediction adsorption ability of an adsorbent. Figure 5 revealed that acidic points on adsorbent's surface were mostly neutralized after 8 h by NaOH 0.018 M solution. When reaction time was kept to 24 h, the difference in NaOH use was tiny. The quantity of acidic points on surface of the adsorbent using Boehm titration was calculated as $1.74 .10^{21}$ $point.g^{-1}$. This result was compared to the study of Dai (2000), and the difference was presented in Table 2. It can be seen that the amount of acidic points on surface of the adsorbent was 10 times higher than that of commercial activated carbon, and approximately 1.5 times compared to that of commercial activated carbon associated with HNO_3 concentrated treatment. It illustrated that phosphoric acid helps to generate more acidic points on surface of carbon than other acids do. Finally, high amount of acidic point on surface of the adsorbent in this study could predictably benefit the adsorbent in adsorption ions in aqueous solution. Surface charge of an adsorbent is an important factor allowing to predict adsorption ability of an adsorbent at different pH solutions. Point of zero charge of the adsorbent was pH 5.7, which can be observed in Figure 6. In other words, the adsorbent could adsorb negatively charged solutes as pH solution was lower than 5.7, and adsorbed positively charged solutes as pH solution was higher than 5.7.

Table 2: Quantity of acidic points on surface of the adsorbents

	Acidic point ($point.g^{-1}$)
Adsorbent (this work)	$1.74 .10^{21}$
Activated carbon + $HNO_{3\ conc.}$ (heated in 4 h)	$1.68 .10^{21}$
Activated carbon + $HNO_{3\ conc.}$ (heated in 2 h)	$1.20 .10^{21}$
Commercial activated carbon	$1.20 .10^{20}$

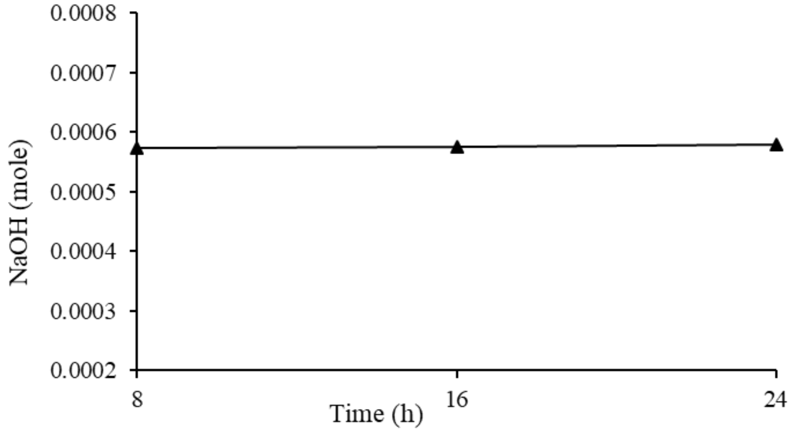


Figure 5: Boehm titration of the adsorbent

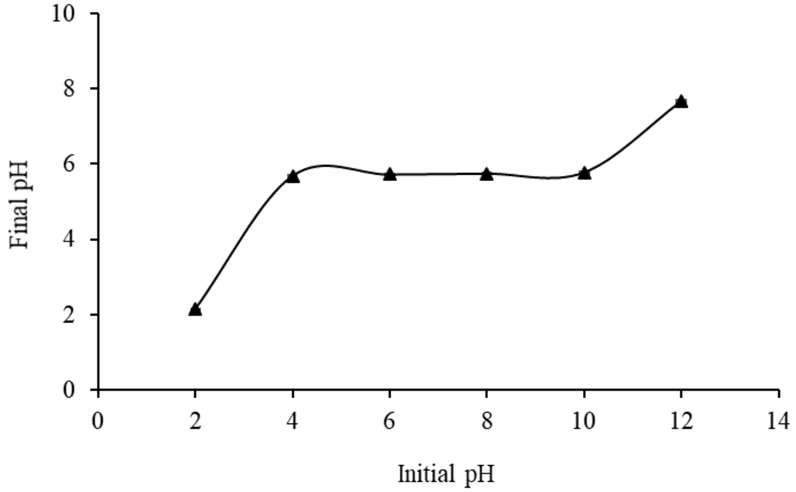


Figure 6: Potentiometric titration of the adsorbent

3.1 Cr(VI) ion adsorption

pH plays a vital role in adsorption of ions in aqueous solution because it determines speciation of solutes in aqueous solution. Effects of pH solution on adsorption yield were presented in Figure 7. When pH was at 1.0, Cr(VI) ions removal was 99.62%. It slightly decreased to 95.23% when pH increased to 2.0. The decrease became more significant when pH continuously went to neutral point. In fact, the yield decreased by 53.89%, 65.76%, 80.25, and 79.87% when pH was respectively at 3.0, 4.0, 5.0 and 6.0. The lowest removal yield (8.71%) was observed at pH 8.0. Those observations can be caused by the fact that increase in pH leads to reduce acidic points on surface of the adsorbent, and rise the quantity of OH group in the solution as well. Moreover, based on speciation of Cr(VI) ion presented in Figure 8, Cr(VI) ion was dominant in $Cr_2O_7^{2-}$ form, and small amount of $HCrO_4^-$ species was also observed in the pH range of 1.0-5.6. After pH 5.6, the amount of $Cr_2O_7^{2-}$ species decreased while that of CrO_4^{2-} increased, and both of them obtained the same quantity at pH 7.2. The CrO_4^{2-} species became more significant when pH was higher. This species reached 100% at pH 9.5. From the above information, one $Cr_2O_7^{2-}$ species needed two acidic points on surface of the adsorbent, but as looking carefully there were two Cr elements in $Cr_2O_7^{2-}$ species; thus, one Cr element adsorbed on one acidic point of the adsorbent's surface. On the other hand, when pH was higher than 5.6, CrO_4^{2-} appeared. Although both species possess two negative charges, CrO_4^{2-} contains only one Cr element while the other consists of two Cr elements. Another important reason was that pH_{pzc} of the adsorbent is 5.7. The surface charge of the adsorbent became less positive when pH increased from 1.0 to 5.7. After this point, the surface charge was more negative and became dominant in alkaline pH solution. From all above viewpoints, the change of pH solution from acidic to basic range caused the decrease in Cr(VI) ions adsorption on surface of the adsorbent.

Effects of contact time on Cr(VI) ion adsorption were presented in Figure 9. Cr(VI) ions were mostly removed after 90 min. Approximate 73% of Cr(VI) was adsorbed in the first 3 min. The removal yield increased to 95.23% when contact time was 20 min, and then slightly rose to 96.30%, 98.95%, and 99.23% after 30, 60, and 90 min adsorption, respectively. Based on the above result, kinetics of Cr(VI) adsorption was calculated and presented in Figure 10 and Table 3. Compared to Pseudo-first-order kinetic equation, Pseudo-second-order kinetic equation presented a good agreement to experimental results. In fact, equilibrium adsorption capacity in Pseudo-second-order equation almost equaled to practical one (331.51 $mg.g^{-1}$) presented in Table 3. Besides, $R^2 = 1$ was a strong illustration for this agreement. From the Pseudo-second-order kinetic equation, it can be seen that the slope is small (a = 0.0199). In other words, ratio t/q_t was low or q_t is high. High q_t means a significant adsorption capacity of an adsorbent. It again confirmed that the adsorbent carbonized from CCP possesses a high adsorption capacity to Cr(VI) ions in aqueous solution at acidic pH solution.

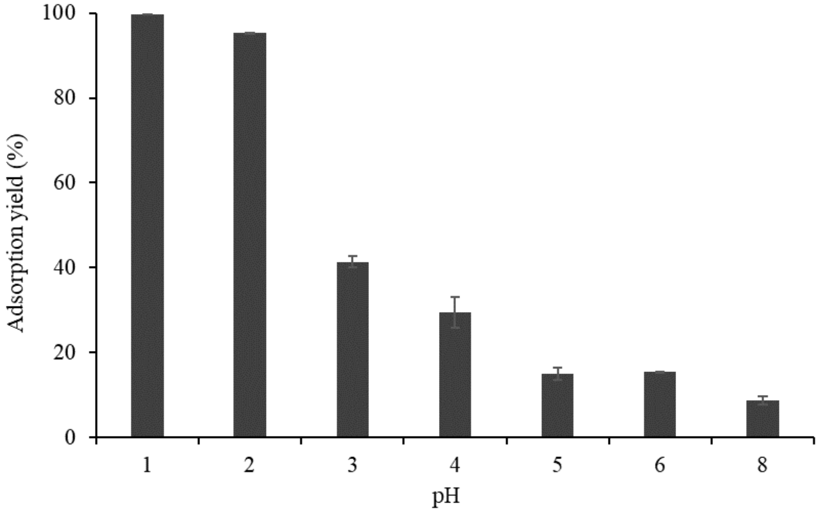


Figure 7: Effects of pH on Cr(VI) ions adsorption [ions concentration = 100 mg.L-1; amount of adsorbent = 0.2 g; contact time = 20 min; stirring speed = 180 rpm; temperature = 25°C]

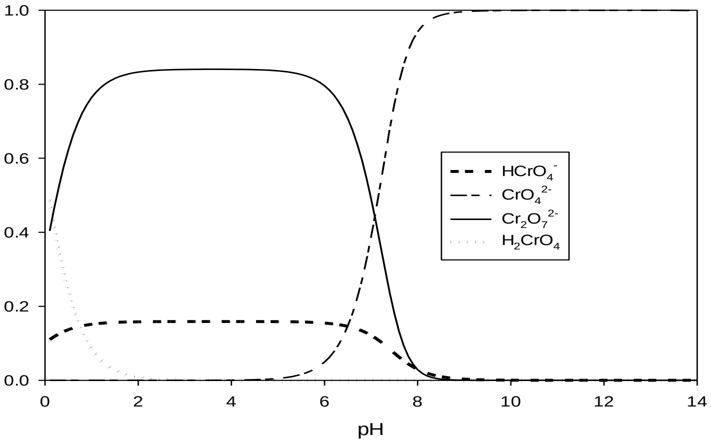


Figure 8: Speciation diagram of Cr(VI) ions with pH variation [2]

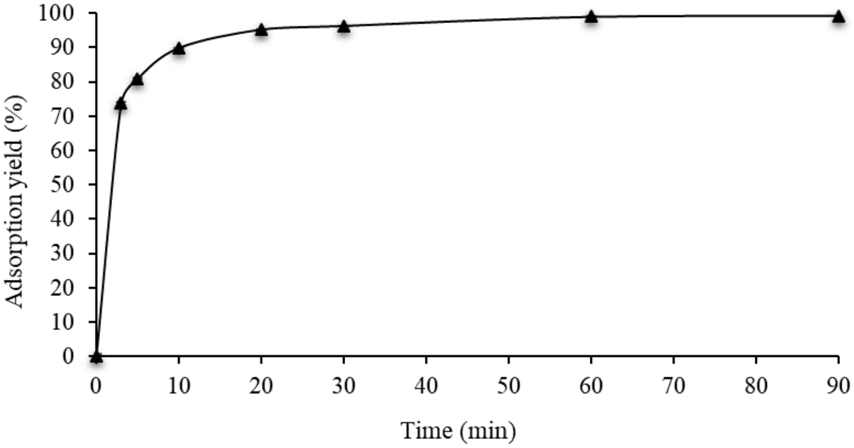


Figure 9: Effects of contact time on Cr(VI) ions adsorption [ions concentration = 100 mg.L^{-1}; amount of adsorbent = 0.2 g; pH 2.0; stirring speed = 180 rpm; temperature = 25°C]

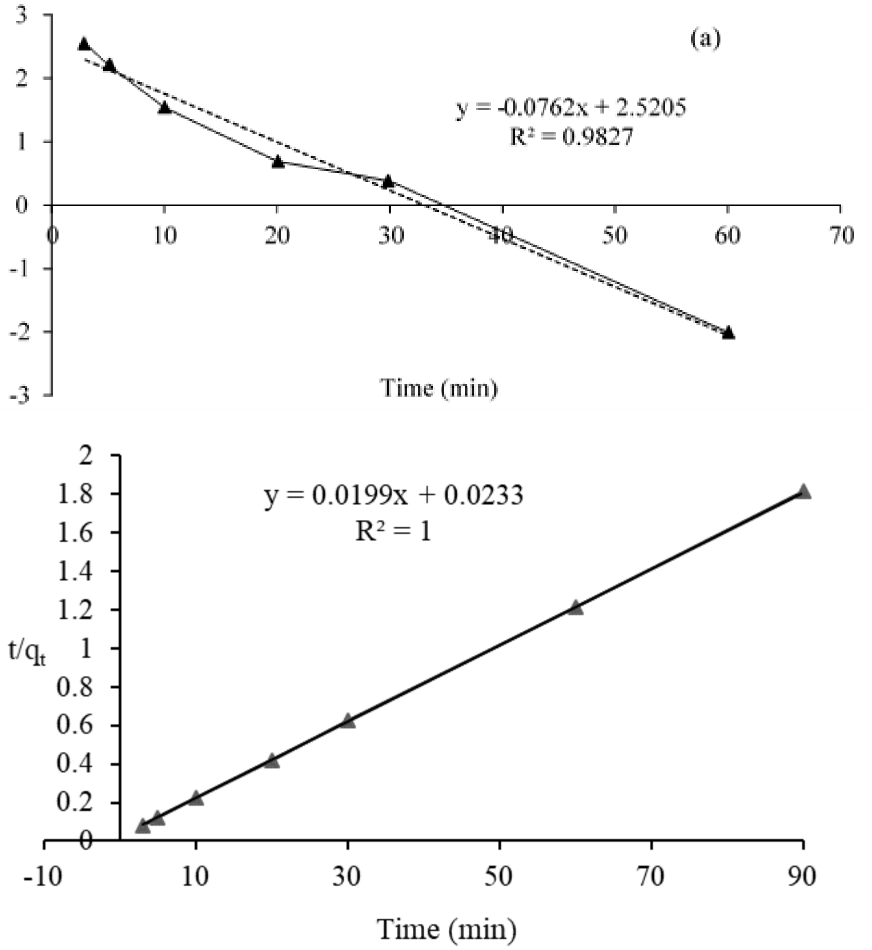


Figure 10: Kinetic plot for Cr(VI) ions adsorption: (a) Pseudo-first-order, (b) Pseudo-second-order

Table 3: Kinetic parameters for Cr(VI) ions adsorption

	a	**b**	**q_e ($mg.g^{-1}$)**	**k_1 ($1.min^{-1}$)**	**k_2 ($g.mg^{-1}.min^{-1}$)**	**R^2**
Pseudo-first-order	-0.0762	2.5205	331.51	-0.0762	-	0.9827
Pseudo-second-order	0.0199	0.0233	50.25	-	0.0174	1

Table 4: Effects of initial concentrations on Cr(VI) ions adsorption

C_0 ($mg.L^{-1}$)	**C_e ($mg.L^{-1}$)**	**Yield (%)**	**q_e ($mg.g^{-1}$)**
50	0.40	99.20±0.01	24.80
75	1.03	98.97±0.05	36.99
100	4.77	95.23±0.05	47.62
125	9.53	92.37±0.28	57.73
150	17.56	88.20±0.44	66.22
175	23.57	86.53±0.39	75.72
200	34.36	82.82±0.53	82.82
225	53.93	76.03±0.65	85.54
250	58.25	76.70±0.36	95.87
275	71.56	73.98±0.29	101.72
300	78.33	73.89±0.48	110.83

Initial concentration of Cr(VI) ions showed a moderate influence on adsorption performance of the adsorbent, and the results were revealed in Table 4. From Table 4, an increase of initial concentration of Cr(VI) ions caused the relative decrease in adsorption yield, but the remarkable increase in adsorption capacity. When C_0 was 50 $mg.L^{-1}$, the yield and q_e were 99.20% and 24.80 $mg.g^{-1}$, respectively. The yield decreased to 95.23% as C_0 was 100 $mg.L^{-1}$ while q_e increased to 47.62 $mg.g^{-1}$ at the same initial concentration of Cr(VI) ions. A slight decrease could be observed in yield when C_0 was varied from 100 to 200 $mg.L^{-1}$ meanwhile q_e showed a notable increase at identical condition. When C_0 continuously increased to 300 $mg.L^{-1}$, both yield and q_e moderately varied to 73.89% and 110.83 $mg.g^{-1}$, respectively. From the above observations, it can be stated that adsorption capacity continuously increased when increasing initial concentration of Cr(VI) ions while the amount of adsorbent was kept unchanged. It implied that Cr(VI) ions adsorbed on the surface of adsorbent via multi-layer adsorption.

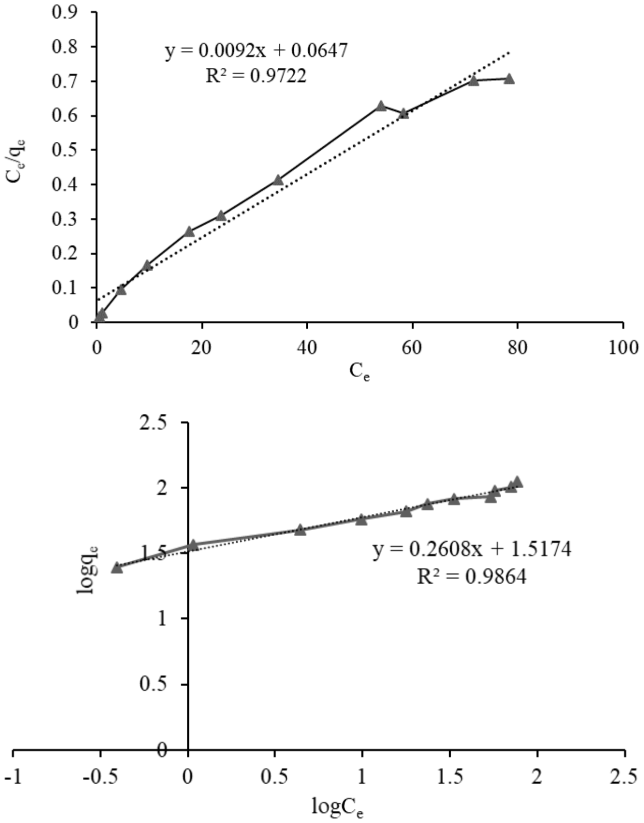


Figure 11: Adsorption isotherms plot of Cr(VI) ions (a) Langmuir, (b) Freundlich

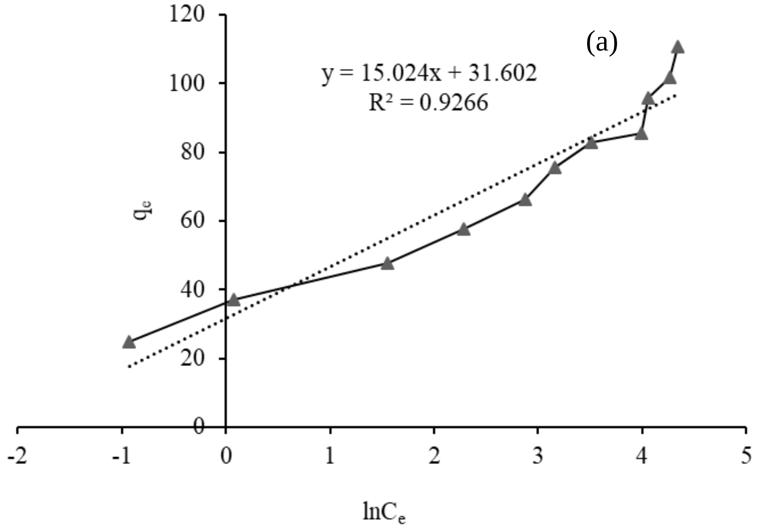
Adsorption isotherms of Cr(VI) ions on surface of the adsorbent were presented in Figure 11. The Freundlich isotherms model well described the experimental data in comparison of Langmuir isotherms model. In fact, R^2 of the Freundlich isotherms model was 0.986 while that of Langmuir isotherms model was 0.972. This again confirmed that multi-layer adsorption is the best model to describe adsorption of Cr(VI) ions by using the adsorbent carbonized from CCP.

To evaluate potential application of the adsorbent carbonized from CCP to remove Cr(VI) ions from aqueous solution, Table 5 might help to clarify the concern. As can be seen from Table 5, the red mud activated by sulfuric acid showed the worse Cr(VI) ions adsorption capacity, 2.34 mg.g^{-1}. Similar adsorption capacities can be found with cow dung activated by H_2SO_4 and CCP activated by H_2SO_4. RHA and tea leaves showed a remarkable increase in Cr(VI) ions adsorption capacity, namely 59.52 and 52.08 mg.g^{-1} respectively. In the comparison of such adsorbents, the adsorption capacity in this study was 2 times higher than these of RHA and tea leaves, 12 times higher than fern, and 35 times higher than CCP activated by H_2SO_4. It can be said that CCP activated by H_3PO_4 prepared in this study presented a high potential in adsorption of Cr(VI) ions.

Table 5: Adsorption capacity of adsorbents derived from various agricultural by-products

Raw materials	Activated agents	Activated conditions	q_{max} (mg.g^{-1})	References
RHA	HCHO	30ºC, 5 h	59.52	Tinh (2011)
Tea leaves	KOH	-	52.08	*Huong et al. (2016)*
Red mud	H_2SO_4	80ºC, 1 h	2.34	Dung *et al.* (2015)
Cow dung	H_2SO_4	120ºC, 24 h	4.50	Das *et al.* (2000)
CCP	H_2SO_4	80ºC, 12 h	3.46	Selvi *et al.* (2010)
CCP	H_3PO_4	200ºC, 6 h	110.83	This work

To understand nature of Cr(VI) ions adsorption by CCP activated by H_3PO_4, Temkin and D-R models were employed and the results are presented in Figure 12. The Temkin constant was calculated as 0.1621 kJ.mol^{-1} in Figure 12 (a). This low value presented a weak interaction between adsorbent and adsorbed, which can be considered as physical adsorption. The bonding energy between Cr(VI) ions and the calculated adsorbent via D-R model was 1.955 kJ.mol^{-1}. This value was smaller than 8 kJ.mol^{-1}, meaning that the interaction between adsorbent and adsorbed in this study was physical interaction. Influences of temperature on Cr(VI) ions adsorption were shown in Figure 13. The adsorption efficiency in Figure 13 slightly increased from 92.23 to 99.16% when temperature increased from 25 to 35°C. The change in efficiency was almost zero as long as temperature continuously increased from 35 to 60°C. It again determined that the main mechanism of Cr(VI) ions adsorption in this study is physical interaction.



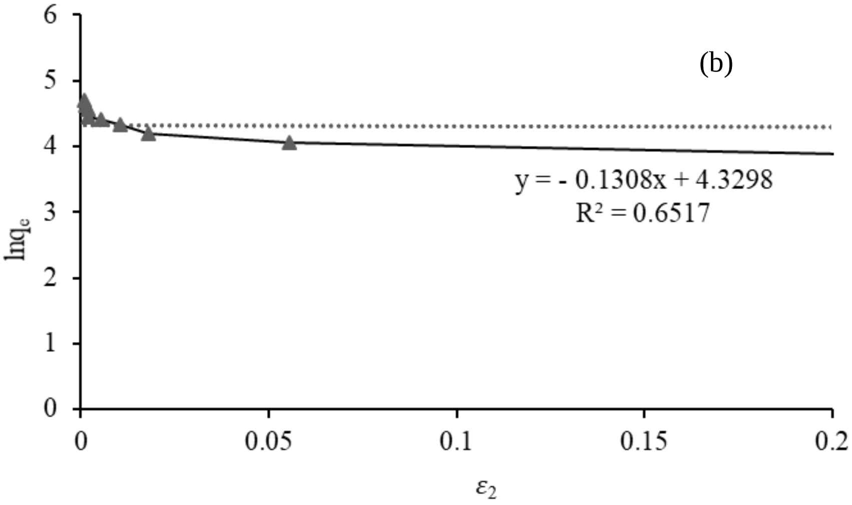


Figure 12: Sorption models for adsorption of Cr(VI) ions (a) Temkin (b) D–R

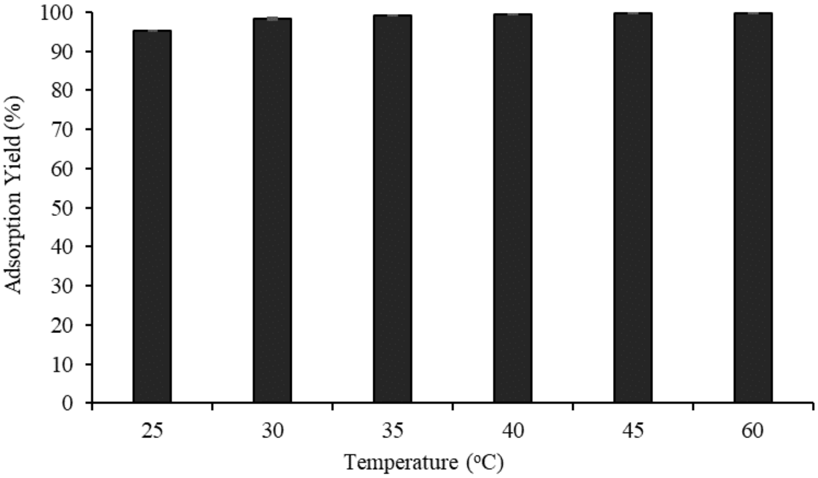


Figure 13: Effects of temperature on Cr(VI) ions adsorption [ion concentration = 100 mg.L^{-1}; amount of adsorbent = 0.2 g; pH 2.0; stirring speed = 180 rpm; contact time = 20 min]

4 CONCLUSIONS

In this study, coconut coir pith was partially carbonized at temperature of 200°C for 6 h with calcination yield of 52.54%. The adsorbent obtained possessed moderate surface area (48.56 m^2.g^{-1}) with pore size of 10.2 nm, but high acidic points on the surface (1.74 .10^{21} point.g^{-1}). The result of potentiometric titration showed that the surface of adsorbent was positive at pH lower than 5.7, and negative at pH higher 5.7. The adsorbent was then used to adsorb Cr(IV) ions in aqueous solution. Adsorption results showed that 0.2 g of adsorbent adsorbed 95.23% of Cr(IV) in the solution containing 100 mgCr.L^{-1} at pH 2.0 within 20 min at 25°C. Adsorption isotherm of Cr(IV) ions and adsorption kinetics of Cr(VI) ions in aqueous solution respectively obeyed Freundlich isotherms model and Pseudo-second-order kinetic equation.

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