

Biosorption of Chromium (VI) Using Rice Husk Ash and Modified Rice Husk Ash

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Abstract: The purpose of this research was to investigate the adsorption ability and determine the suitable condition for adsorption of Chromium (Cr(VI)) in synthetic wastewater by Rice Husk Ash (RHA) and 0.1 NaOH modified RHA (MRHA). Adsorption abilities of the adsorbents in various conditions were examined in batch experiment. Contact time, pH, adsorbent dose and initial Cr(VI) concentration were varied to perform the adsorption conditions. The results indicated that all adsorption parameters affected to the adsorption of Cr(VI) by RHA and MRHA. At the suitable condition (pH 2, contact time 180 min, room temperature, initial Cr(VI) concentration 10 mg L⁻¹, adsorbent dose 40 g L⁻¹, adsorbent size of 250-500 µm), the adsorption abilities of RHA and MRHA were 0.49 and 0.84 mg g⁻¹ with removal efficiencies of 59.12 and 81.39%, respectively. Langmuir isotherm was found to better fit the experiment data rather than Freundlich isotherm. As waste-recycle products, RHA and MRHA has been proved to be an effective adsorbent for Cr(VI) removal.

Key words: Rice husk ash, Cr(VI), modification, adsorption isotherms, adsorption parameters, Thailand

INTRODUCTION

Chromium in the hexavalent form Cr(VI) is a significant heavy metal is widely used in various industries such as leather tanning, electroplating, metal processing and wood preservatives including paint and pigment (Raji and Anirudhan, 1997; Garg *et al.*, 2004). Cr(VI) poisoning in humans targets the nasal septum (Khatoon *et al.*, 2009) and can causes lung cancer (Hughes *et al.*, 1994) and skin ulcerations (McCarron *et al.*, 2000). As a result, many researchers have tried to determine cost effective and economic method for removal of this chemical. Well established methods include filtration, chemical precipitation, adsorption, electrodeposition and membrane systems and an ion exchange process.

There are drawbacks to these methods. The chemical precipitation and reduction methods requires additional separation techniques for the treatment and disposal of the high quantities of waste metal residual sludge that is produced. These additional separation techniques use a lot of treatment chemicals and the residual Cr(VI) concentration required in the treated wastewater is not achieved because of the structure of the precipitates. The application of membrane systems for wastewater treatment has major problems like membrane scaling, fouling and blocking. The ion exchange process has a

drawback in the high cost of the resin, while the electrodeposition method is more energy intensive than other methods. Among these methods adsorption is one of the most economically favorable and a technically simple (Karthikeyan *et al.*, 2005). Conventionally, activated carbons have been found to be an effective adsorbents for recovering Cr(VI) from wastewater (Aggarwal *et al.*, 1999).

However, the process is expensive and uses high amount of energy in its production, which has led to the search for new strategies for developing low-cost materials with a good capacity for Cr(VI) removal (Saueprasearsit *et al.*, 2010; Guo *et al.*, 2009; Karthikeyan *et al.*, 2005; Khezami and Capart, 2005), such as bagasse fly ash (Gupta and Ali, 2004), rice bran (Singh *et al.*, 2005), maple saw dust (Yu *et al.*, 2003), coconut husk, palm pressed fibers (Tan *et al.*, 1993) and various other low-cost biological wastes. Rice husk is an important Thai agricultural waste which is produced in high quantity each year.

It is usually used as a fuel or a raw material in many industries such as fertilizer manufacturing and bio-coal production including biomass power plant. Its utilization as a fuel produces rice husk ash which may cause damage to human health and the environment, such as occurring particulate matter, destroying good landscape, etc. Consequently, rice husk ash which is an industrial waste

from the Roi-Et Green Power Plant biomass power plant in Roi-Et, Thailand was used as an adsorbent in this research. This research investigated the mechanism of Cr(VI) adsorption, adsorption capacity and adsorption isotherm of rice husk ash and modified husk ash. The MRHA was developed to enhance of the adsorption capacity of rice husk ash. The effects of adsorption parameters as pH, contact time and initial Cr(VI) concentration were examined.

Preparation of Rice Husk Ash (RHA): The RHA from Roi-et Green Power Plant was washed with distilled water until the pH was constant, dried in an oven at 105°C for 24 h. It was cooled in incubator and determined its size distribution by USA Standard Sieve (≤ 125 , 125-250, 250-500, 500-600, 600-710 and 710-850 μm). The size distribution results are shown in Table 1. Thus, studied particle size was 250-500 μm . Finally, RHA was size separated and stored in plastic containers for the future experiments.

Preparation of Modified Rice Husk Ash (MRHA): The modification of rice husk ash done by, the RHA was mixed with 0.1 M NaOH (1:20) for 30 min, after treatment with 0.1 M NaOH, the excess NaOH form the Modified Rice Husk Ash (MRHA) was washed with distilled water until the pH was constant and then it was dried in an oven at 105°C for 24 h.

Preparation of synthetic Cr(VI) wastewater: Aqueous solutions (1,000 mg L^{-1}) of Cr(VI) was prepared by dissolving $\text{K}_2\text{Cr}_2\text{O}_7(\text{s})$ in distilled water and diluted to get the desired concentration as 5-30 mg L^{-1} . The Cr(VI) concentrations were measured by Atomic Adsorption Spectrophotometer (AAS) (AA-6200, Shimadzu).

Effects pH, contact time and initial Cr(VI) concentration

Effect of pH: The effect of system pH was studied by varying the initial pH (2-7) of 50 mL Cr(VI) solutions at concentration 10 mg L^{-1} by adjusting the pH with 0.1 M NaOH and 0.1 M HCl. The RHA dosage of 10 g L^{-1} was mixed with the solutions in a 250 mL flask and then shaking the mixtures at 120 rpm and the room temperature. After shaking the flasks for 30 min, the reaction mixtures were separated by filtering. Then, the residue Cr(VI) in the filtered solutions were measured by AAS. In addition, the pH of aqueous solution was also analyzed by the pH meter (Classic g 24, Scientific).

The absorbent was changed from RHA to be MRHA for studying the effect of pH on Cr(VI) removal by MRHA. The percent of Cr(VI) adsorption was determined by the following equation:

Table 1: Size distribution of rice husk ash

Sieve number	Size (μm)	Quantity (g)	By weight (%)
270	≤ 125	38.92	19.78
120	125-250	46.08	23.42
60	250-500	60.21	30.60
35	500-600	14.49	7.36
30	600-710	13.72	6.97
25	710-850	23.31	11.84
Total		196.73	100.00

$$\text{Cr(VI)adsorption(\%)} = \left[\frac{C_0 - C_e}{C_0} \right] \times 100 \quad (1)$$

where, C_0 and C_e are the initial and equilibrium concentration (mg L^{-1}), respectively.

Effect of contact time: The experiments were carried out by using 50 mL Cr(VI) solution at 10 mg L^{-1} which adjusted pH to the optimal pH. The RHA dosage of 10 g L^{-1} was mixed with the solutions in a 250 mL flask and then shaking the mixtures at 120 rpm and the room temperature. After shaking the flask for 0, 10, 30, 60, 180 and 360 min, the reaction mixtures were separated by filtering.

Then, the residue Cr(VI) in the filtered solutions were measured by the AAS and pH meter, respectively. The absorbent was changed from RHA to the MRHA for studying the effect of contact time on Cr(VI) removal by MRHA.

Effect of adsorbent dosage: The experiments were carried out by using 50 mL Cr(VI) solution at concentrations 10 mg L^{-1} which adjusted pH to the optimal pH. The RHA dosage (10, 20, 30, 40 and 50 g L^{-1}) was mixed with the solutions in a 250 mL flask and then shaking the mixtures at 120 rpm and the room temperature. After shaking the flask for the suitable contact time, the reaction mixtures were separated by filtering. Then, the residue Cr(VI) in the filtered solutions were measured by the AAS and pH meter, respectively. The absorbent was changed from RHA to the MRHA for studying the effect of initial Cr(VI) concentration on Cr(VI) removal by MRHA.

Effect of initial Cr(VI) concentration: The experiments were carried out by using 50 mL Cr(VI) solution at concentrations 10-30 mg L^{-1} which adjusted pH to the optimal pH. The optimal RHA dosage was mixed with the solutions in a 250 mL flask and then shaking the mixtures at 120 rpm and the room temperature. After shaking the flask for the appropriate contact time, the reaction mixtures were separated by filtering. Then, the residue Cr(VI) in the filtered solutions were measured by the AAS and pH meter, respectively. The absorbent was changed from RHA to the MRHA for studying the effect of initial Cr(VI) concentration on Cr(VI) removal by MRHA.

Sorption isotherms (Guo *et al.*, 2009): Adsorption isotherms describe how adsorbates interact with adsorbents and are critical in optimizing the use of adsorbents. Adsorption efficiency is commonly determined by the extent of solute adsorbed from the solution. The amount of adsorbate per unit mass of adsorbent at equilibrium (q_e , mg g⁻¹) and the adsorbate equilibrium concentration (C_e , mg L⁻¹) allows plotting the adsorption isotherm versus and the mathematical models can be used to describe and characterize the adsorption process. In this research, two isotherm models were used to study the equilibrium sorption as Langmuir and Freundlich models. The Langmuir model assumes that the uptake of metal ions on a homogenous surface by monolayer adsorption without any interaction between adsorbed ions. The Langmuir model for equilibrium ion removed is given by:

$$q_e = \frac{q_{\max} b C_e}{(1 + b C_e)} \quad (2)$$

where, q_e is the metal ions concentration on the adsorbent at equilibrium (mg of metal ion/g of adsorbent), q_{\max} is the maximum metal uptake per unit mass of adsorbent (mg g⁻¹). b is Langmuir constant (L mg⁻¹) related to energy of sorption which reflects quantitatively the affinity between the adsorbent and metal ions. The values of q_{\max} and b are the characteristics of the Langmuir model. They can be determined by linearizing Eq. 2 as shown in Eq. 3:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} b} + \frac{C_e}{q_{\max}} \quad (3)$$

Therefore, a plot of C_e/q_e versus C_e gives a straight line of slope $1/q_{\max}$ and intercept $1/q_{\max} b$ (Fig. 1 and 2), which shows that the adsorption of Cr(VI) follow by RHA and MRHA Langmuir isotherm model. The Langmuir parameters, q_{\max} and b are calculated from the slope and intercept of the graphs and are shown in Table 1.

On the other hand, the Freundlich model is chosen to evaluate parameters associated to the sorption behavior. The equation is commonly represented by:

$$q_e = K_f C_e^{1/n} \quad (4)$$

where, K_f and n are Freundlich constants, indicating the adsorption capacity and the adsorption intensity respectively. The above equation is rearranged in linear form to give:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (5)$$

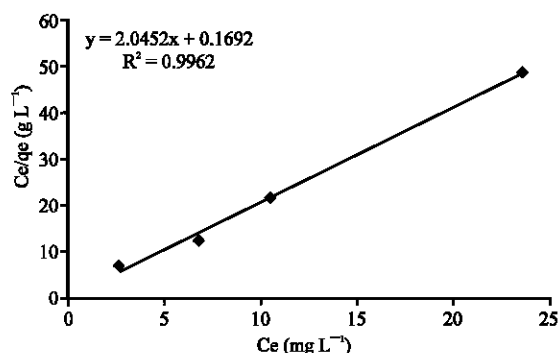


Fig. 1: Langmuir isotherm for the adsorption of Cr(VI) by RHA

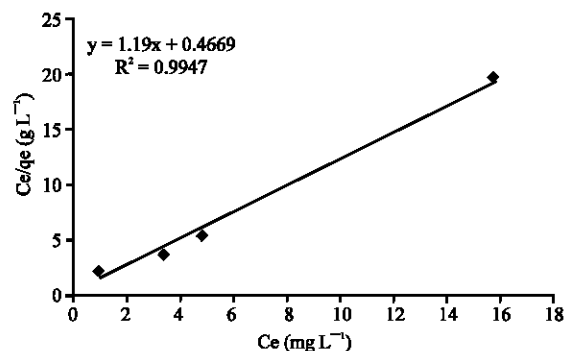


Fig. 2: Langmuir isotherm for the adsorption of Cr(VI) by MRHA

Table 2: Langmuir and Freundlich isotherm constants for adsorption of Cr(VI) by RHA and MRH

Adsorbent	Langmuir isotherm			Freundlich isotherm		
	q_{\max} (mg g ⁻¹)	b (l mg ⁻¹)	R^2	K_f (mg g ⁻¹)	n (-)	R^2
RHA	0.49	12.09	0.9962	0.39	11.20	0.3495
MRHA	0.84	2.55	0.9947	0.50	4.08	0.6363

K_f and n are, respectively, determined from the intercept and slope of plotting in q_e versus in C_e . The Freundlich parameters for the adsorption of Cr(VI) are also shown in Table 2.

RESULTS AND DISCUSSION

Effect of pH on adsorption: The pH of the solutions has been identified as the most important variable governing metal adsorption. This is partly due to the fact that hydrogen ions themselves are strong competing ions and partly that the solution pH influences the chemical speciation of the metal ions as well as the ionization of the functional groups onto the adsorbent surfaces (Kadirvelu and Namasivayam, 2003). Effects of pH on the

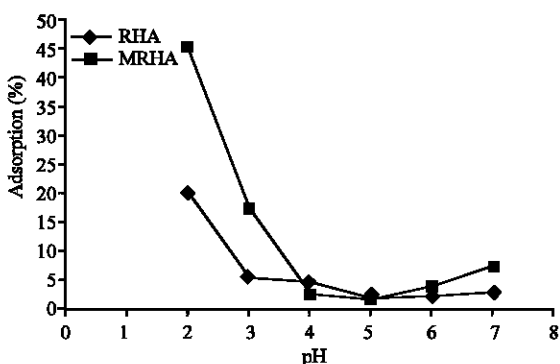


Fig. 3: Effect of pH on adsorption of Cr(VI) by RHA and MRHA

uptake capacities of Cr(VI) on RHA and MRHA were investigated. Experimental studies were carried out at the room temperature with initial Cr(VI) concentration 10 mg L^{-1} using adsorbent dosage (adsorbent size $250\text{-}500 \text{ }\mu\text{m}$) 10 g L^{-1} , agitation speed 120 rpm for 30 min . Moreover, hydroxide ions (OH^- ions) in the solution can bind with Cr(VI) ions to hydroxide complexes form solid so the system pH should not exceed 7 (2.0-7.0). The adsorption results are shown in Fig. 3.

From Fig 3, the results show that the Cr(VI) adsorption decreased with an increase of pH. The maximum value of Cr(VI) retention occurred approximately at pH 2.0. At lower pH, the biosorbent is positively charged due to protonation and dichromate ion exists as anion leading to an electrostatic attraction between them (Wasay *et al.*, 1999). As pH is increases, deprotonation starts and thereby results in decrease of adsorption capacity. Cr(VI) forms stable complexes such as $\text{Cr}_2\text{O}_7^{2-}$, HCrO_4^- , CrO_4^{2-} and HCrO_7^- depending on the pH of the solution. The fraction of any particular species depends upon the chromium concentration and pH of the solution (Namasivayam and Kadirvelu, 1999; Lee *et al.*, 2005; Umesh *et al.*, 2007). Moreover, adsorption of Cr(VI) by MRHA has been found to be higher than RHA. The fact that a base treatment can be remove surface impurities and expose available binding sites for metal bioadsorption after pretreatment, might be the reason for the increase in metal biosorption (Bhatti *et al.*, 2007; Gerdoa-Torresdey *et al.*, 2000).

Effect of contact time on adsorption: The rate of adsorption that takes place is one of the parameters for economical wastewater treatment plant application. Consequently, it is important to establish the time dependence of such systems under various process conditions (Patnukao *et al.*, 2008). Effects of contact time on the removal of Cr(VI) are shown in Fig. 4. Experiment studies were carried out at the room temperature with

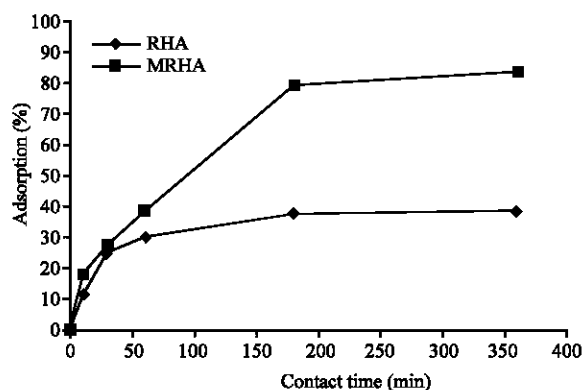


Fig. 4: Effect of contact time on adsorption of Cr(VI) by RHA and MRHA

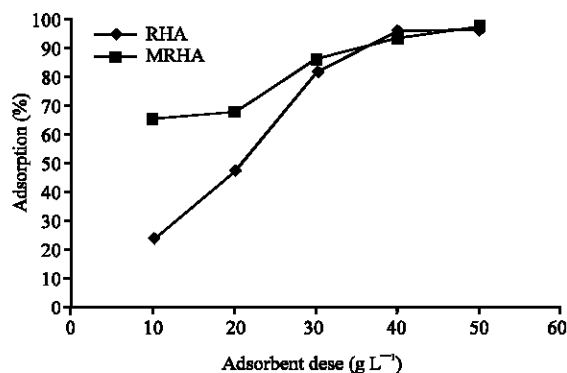


Fig. 5: Effect of adsorption dose on adsorption of Cr(VI) by RHA and MRHA

initial Cr(VI) concentration 10 mg L^{-1} using adsorbent dosage (adsorbent size $250\text{-}5600 \text{ }\mu\text{m}$) 10 g L^{-1} at pH 2.0 and agitation speed 120 rpm for contact time 0, 10, 30, 60, 180 and 360 min.

In Fig. 4, it shows that the rate of Cr(VI) adsorption is found to be very rapid gradually increase in the period time of 0-180 min and thereafter, the removal of Cr(VI) ions is almost constant. During the initial stage of sorption, a large number of vacant surface sites are available for adsorption. After a lapse of some time, the remaining vacant surface sites have difficulty in becoming occupied due to repulsive forces between the adsorbate molecules on the solid surface and in the bulk phase. Besides, the metal ions are adsorbed into the mesopores that get almost saturated with Cr(VI) ions during the initial stage of adsorption (Lu *et al.*, 2009). In this study, the appropriate contact time for Cr(VI) adsorption on RHA and MRHA is 180 min.

Effects of absorbent dose on adsorption: Effects of adsorbent dose on the removal of Cr(VI) are shown in Fig. 5. Experiment studies were carried out at the room

temperature with Cr(VI) initial concentration 10 mg L^{-1} using adsorbent dosage (adsorption size $250\text{-}500 \text{ }\mu\text{m}$) 10, 20, 30, 40 and 50 g L^{-1} at pH 2.0 and agitation speed 120 rpm for 180 min.

Figure 5 shows the adsorption of Cr(VI) as a function of RHA and MRHA dosages. It is apparent that increasing the adsorbent dose causes an increase in the amount of Cr(VI) but a decrease the amount adsorbed per unit mass, adsorption density. It is readily understood that the number of available adsorption sites increases by increasing the adsorbent dose and it, therefore, results in the increase of the amount of adsorbed Cr(VI). The decrease in adsorption density with increase in the adsorbent dose is mainly because of unsaturation of adsorption sites through the adsorption process (Yu *et al.*, 2003; Pehlivan *et al.*, 2008). Another reasons may be due to the particle interaction, such as aggregation, resulting from high adsorbent dose. Such aggregation would lead to decrease in total surface area of the adsorbent and an increase in diffusion path length (Shukla *et al.*, 2002). The results can be concluded that the appropriate adsorbent dose is 40 g L^{-1} . After this dose, the increasing rate of Cr(VI) removal is almost constant.

Effect of initial Cr(VI) concentration: Effects of initial Cr(VI) concentration on the removal of Cr(VI) was shown in Fig. 6. Experiment studies were carried out at the room temperature with Cr(VI) initial concentration 10, 15, 20 and 30 mg L^{-1} using adsorbent dosage (adsorption size $250\text{-}500 \text{ }\mu\text{m}$) 40 g L^{-1} at pH 2.0 and agitation speed 120 rpm for 180 min.

The effect of initial Cr(VI) concentration is also examined (Fig. 6). In the case of low concentrations, the ratio of the initial number of moles of Cr(VI) ions to the available surface area is larger and subsequently the fractional adsorption becomes independent of initial concentrations. The suitable initial Cr(VI) concentration is 10 mg L^{-1} . At higher concentrations, the available sites of adsorption become decreased and hence the percentage adsorption of Cr(VI) decreases. It is readily understood that the number of available adsorption sites are inconstant but increasing the adsorbate.

Moreover, this research was repeated the adsorption capacity of RHA and MRHA at the optimal condition as pH 2, contact time 180 min, the room temperature, adsorbent size $250\text{-}500 \text{ }\mu\text{m}$, agitation speed 120 rpm, adsorbent dose 40 g L^{-1} and initial Cr(VI) concentration 10 mg L^{-1} . In this condition, the adsorption ability RHA and MRHA are 59.12 and 81.39%, respectively.

Sorption isotherms: Adsorption isotherms for adsorption of Cr(VI) on RHA and MRHA were calculated from the results of studying effects of initial Cr(VI) concentration.

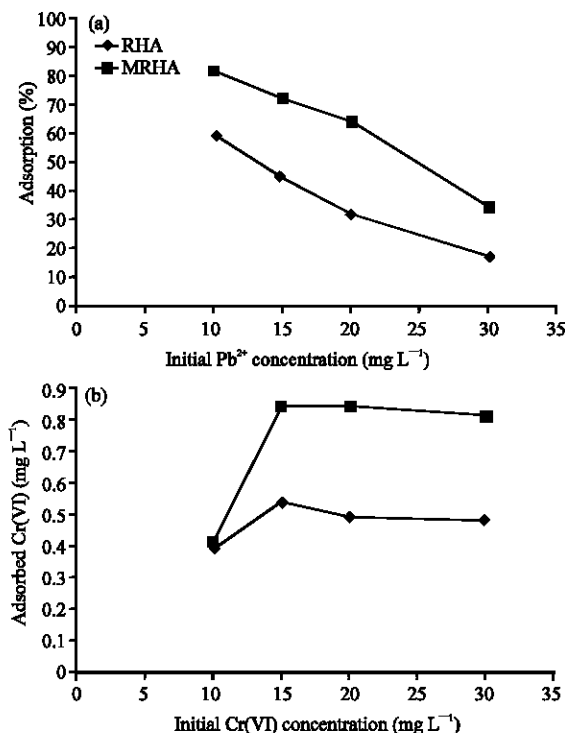


Fig. 6: (a) Effect of initial Cr(VI) concentration on adsorption of Cr(VI) by RHA and MRHA; (b) Effect of initial Cr(VI) concentration to adsorbed Pb^{2+}

Table 3: Comparison between adsorption capacity of RHA and MRHA and other adsorbents

Absorbent	Adsorbent capacity (mg g^{-1})	References
Soya cake	0.28	Daneshvar <i>et al.</i> (2002)
Maize comcob	0.28	Umesh <i>et al.</i> (2007)
Jatropha oil cake	0.82	Umesh <i>et al.</i> (2007)
Sugar cane bagasse	0.63	Umesh <i>et al.</i> (2007)
Almond shell	0.58	Pehlivan <i>et al.</i> (2008)
RHA	0.49	This study
MRHA	0.84	This study

The results present that the experiment data were better fitted to the Langmuir equation ($R^2_{\text{RHA}} = 0.9962$, $R^2_{\text{MRHA}} = 0.9947$) than the Freundlich equation ($R^2_{\text{RHA}} = 0.9962$, $R^2_{\text{MRHA}} = 0.9947$) (Table 2).

From the results, the Langmuir adsorption model, based on the sorption on a homogeneous surface by monolayer sorption without interaction between sorbed species becomes more appropriate to describe the process.

Table 3 shows a comparison between the results of this research and others found in the literature. The values of Cr(VI) specific uptake found in this research were higher than the other excepted researchs. Thus, the RHA and MRHA are the effective adsorbents for the Cr(VI) adsorption from aqueous solutions.

CONCLUSION

Rice husk ash is an interesting alternative adsorbent for heavy metals because of its quantity and characteristics. Although, these results are presented that adsorption capacity is low but it is useful for alternative waste management programs and friendly with ecosystems. Moreover, it can increase the effectiveness of Cr(VI) adsorption from RHA by NaOH modification.

The results indicated that all adsorption parameters affected the adsorption of Cr(VI) by RHA and MRHA. The adsorption ability of RHA and MRHA were 59.12 and 81.39% at pH 2, contact time 180 min, the room temperature, adsorbent size 250-500 μm , agitation speed 120 rpm, adsorbent dose 40 g L^{-1} and initial Cr(VI) concentration 10 mg L^{-1} . Furthermore, the results were fitted to Langmuir isotherm better than Freundlich isotherm.

Therefore, the adsorption mechanism based on the sorption on a homogeneous surface by monolayer sorption without interaction between sorbed species becomes more appropriate to describe this process. The adsorption capacity of RHA and MRHA were 0.49 and 0.84 mg g^{-1} , respectively. Finally, the RHA and MRHA utilization is an alternative waste management or environmental conservation. All adsorption parameters as pH, contact time, adsorbent dose and initial Cr(VI) concentration can affect to the Cr(VI) adsorption by RHA and MRHA.

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