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A STUDY ON KINETIC AND THERMODYNAMIC ADSORPTION OF FLUORIDE FROM AQUEOUS SOLUTION ONTO ALUMINIUM HYDROXIDE COATED RICE HUSK ASH

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ABSTRACT

The fluoride adsorption on aluminum hydroxide coated rice husk ash material (RHA/$Al(OH)_3$) was demonstrated in this study. The isothermal data indicated that the Langmuir model well described the adsorption system with the maximum monolayer adsorption capacity of 8.2 $mg.g^{-1}$. The kinetic results revealed that the pseudo-second-order rate model fitted the experiments data better than the pseudo-first-order one. Furthermore, the adsorption of fluoride onto this material may be the chemical adsorption. Thermodynamic parameters (ΔG^o and ΔH^o) in the range of temperature from 30 to 70 ^oC showed that the adsorption was a spontaneous and an endothermic process. RHA/$Al(OH)_3$ could be used for well-treatment of fluoride contaminated well-water sampling in Ninhhoa district (Khanhhoa province). With the initial content of fluoride of 10.1 $mg.L^{-1}$ and after 2 hours of treating with the dose of 4.0 ÷ 7.0 $g.L^{-1}$, the concentration of fluoride in the samples decreased to 0.5 ÷ 1.5 $g.L^{-1}$, that met acceptable limit of WHO.

Keywords: aluminium hydroxide coated rice husk ash, adsorption, fluoride.

1. INTRODUCTION

With human and animals body, fluorine is an integral element which plays the role of regulating the metabolism of calcium and phosphorus. It is necessary for the development of teeth and bone, the formation of teeth's tusk and enamel. When we lack of fluorine, our teeth will be decay and the bones will be spongy. However, if fluoride concentration exceeds acceptable limit (1.5 $mg.L^{-1}$), the dental fluorosis will appear with main symptoms are that: teeth appearance is marred by discoloration or brown markings and break easily. Besides, the redundancy of fluoride in human body can make the bone weak, get out of shape, break easily, damage to thyroid gland, endocrine, brain… Nowadays, the fluorosis appears widely in over 25 countries and about 62 millions people catch this disease, especially, in Bangladesh, China, Mongolia India… [1]. In Vietnam, fluoride contaminated well-water (3 to 14 $mg.L^{-1}$) results to the fluorosis widespread in Khanhhoa, Phuyen, Quangnam, Thaibinh provinces… [2]. So, the study on the removal of fluoride from running water is an urgent problem. One of methods using for treatment of fluoride interested most is using by sorbents such as hydroxyapatite [3], kaoline [4] or red mud [5] because of its high effect in reality.

In previous paper, the preparation of $RHA/Al(OH)_3$ was studied. The obtained material exhibited a large amount of amorphous silica and activated carbon with fine-grained particles, high porosity and it well adsorbs fluoride.

In this paper, isothermal, kinetic and thermodynamic results of the adsorption of fluoride onto this material were demonstrated.

2. EXPERIMENTS

2.1. Adsorbent

$RHA/Al(OH)_3$ was prepared from rice husk ash and Al^{3+} solution [6]. Rice husk was treated by 1M HCl solution for 24 hours after being burned at 700 ^oC for 60 minutes. Rice husk ash (RHA) was obtained after being washed by deionized water and dried at 100 ^oC. The mixture containing RHA and 0.1 M Al^{3+} solution was adjusted to the pH of 5÷6 and stirred for 30 minutes. The content of Al_2O_3 in obtained material was 20 %. $Al(OH)_3$ would be precipitated and dispersed on the surface of RHA particles. The solid product was washed and dried at 100 ^oC.

The solutions of fluoride were prepared from NaF (PA, Sigma-Aldrich). The concentration of fluoride was determined by molecular absorption spectroscopy with Zirconyl - Alizarin chelate using UV-Vis T80 device (Helios) at the wavelength of 527 nm.

2.2. Effect of pH

With the aim of assessing the effect of pH to the fluoride adsorption, nine samples containing 100 mL fluoride solution were prepared with the concentration of 10 $mg.L^{-1}$. The pH of samples was adjusted from 3 to 11 (with the notations of the samples were from pH3 to pH11), the temperature was fixed at 25 ^oC. Each sample was stirred for 2 hours after adding $RHA/Al(OH)_3$ into the solution with the dose of 5 $g.L^{-1}$ in order to reach to the adsorption equilibrium. The solution was filtered and the fluoride concentration was determined. The efficient of the adsorption was calculated by the equation as follows:

$$H = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

where C_o and C_e are the concentration of fluoride in the solution before and after adsorption, respectively ($mg.L^{-1}$).

2.3. Effect of the dose of $RHA/Al(OH)_3$

The suitable dose of $RHA/Al(OH)_3$ was chosen after investigating the efficient of the fluoride adsorption of 10 samples containing adsorbent with the dose varying from 1 to 10 $g.L^{-1}$

(notations of samples were from LL1 to LL10). Other factors were fixed and the practical steps were described in 2.2 item.

2.4. Adsorption isotherm

The maximum fluoride adsorption capacity (q_m) of RHA/Al(OH)$_3$ was determined by isotherm survey. Seven samples were prepared, each sample containing 100 mL of fluoride solution with the content rising from 5 to 35 mg.L^{-1} (the samples were notated from C5 to C35). The dose of adsorbent was fixed at 5 g.L^{-1}. Other factors were fixed and the practical steps were described in 2.2 item.

In this research, the Langmuir and Freundlich isotherm models [7, 8, 9, 10] were used to evaluated the adsorption.

The Freundlich isotherm model is based on heterogeneous surfaces suggesting that binding sites are not equivalent and independent. The Langmuir isotherm model is applicable to the homogeneous adsorption where the adsorption of each adsorbate molecule onto the surface has an equal adsorption activation energy. The Freundlich and Langmuir models can be expressed as follows:

$$\ln q_e = \ln K_F + \frac{1}{n} C_e \qquad (2)$$

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K \times q_m} \qquad (3)$$

where: C_e is the equilibrium concentration of fluoride in the solution after adsoption (mg.L^{-1}); q_e is adsorption amount of RHA/Al(OH)$_3$ (mg.g^{-1}), that was calculated by equation: $q_e = \frac{(C_o - C_e)V}{m}$, where: C_o is the initial concentration of fluoride (mg.L^{-1}), V is the volume of fluoride solution (L), m is the mass of RHA/Al(OH)$_3$ (g); q_m is maximum fluoride adsoprtion capacity (mg.g^{-1}); K is Langmuir constant which is related to the strength of adsorption; K_F and n is Fruendlich constants that are related to the adsorption capacity and the adsorption intensity.

2.5. Adsorption kinetic

The kinetics data of fluoride adsorption was obtained when the effect of time to adsorption capacity of adsorbent was investigated. RHA/Al(OH)$_3$ was added into 100 mL of 10 mg.L^{-1} fluoride solution with the dose of 5 g.L^{-1}, the temperature was fixed at 25^{o}C. After 10 minutes, 10 mL of the stirring mixture was separated and the fluoride was determined.

The pseudo-first-order rate model (4) and the pseudo-second-order rate model (5) [7, 8, 10] was applied to test the kinetic data.

The pseudo-first-order and pseudo-second-order kinetic equations are given as:

$$\ln(q_e - q_t) = \ln(q_e) - k_1 t \qquad (4)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \qquad (5)$$

where: q_e (mg.g^{-1}) and q_t (mg.g^{-1}) are the amount of fluoride ions adsorbed on the adsorbent at equilibrium and any time, respectively; k_1 (L.s^{-1}) and k_2 (g.mg^{-1}.s^{-1}) are the pseudo-first-order and pseudo-second-order rate constant.

2.6. Thermodynamic studies

In order to understand the mechanism of adsorption, thermodynamic parameters such as ΔG^o, ΔH^o and ΔS^o were calculated according to the study of effect of temperature to adsorption. Five groups of samples were prepared at 5 temperatures varying from 303 K to 343 K. Each group including 10 samples that contained 0.5 g of RHA/$Al(OH)_3$ and 100 mL of fluoride solution corresponding with the initial concentration varying from 10 to 40 $mg.L^{-1}$. The mixtures were stirred for 80 minutes until the adsorption reached equilibrium. After that, fluoride contents of separating solution were determined.

The equilibrium constant of adsorption (K_c) was approximately calculated by the equation from the studies of J. Rahchamani [9]:

$$K_C = \frac{C_{ae}}{C_e} = \frac{q_e}{C_e} \quad (6)$$

where: C_{ae} and C_e are equilibrium fluoride concentration on the adsorbent and solution ($mg.L^{-1}$); ΔG^o parameter of adsorption was determined by (7) equation. From the obtained results, ΔH^o and ΔS^o parameters were obtained from the correlation equation between $\ln K_C$ and 1/T basing on (8) equation.

$$\Delta G^o = -RT \ln K_C \quad (7)$$

$$\ln K_C = -\frac{\Delta G^o}{RT} = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT}. \quad (8)$$

3. RESULTS AND DISCUSSION

3.1. Effect of pH

Fluoride adsorption behavior of RHA/$Al(OH)_3$ under different pH was investigated to determine the optimal pH for removing of fluoride ions. The results are shown in Table 1.

Table 1. The influence of pH on fluoride removal capacity.

Notations	pH3	pH4	pH5	pH6	pH7	pH8	pH9	pH10	pH11
pH	3.0	4.0	4.8	6.0	7.0	8.0	9.0	10.0	11.0
C_e ($mg.L^{-1}$)	1.15	0.69	0.52	0.41	0.42	0.42	0.41	0.40	2.19
H (%)	88.5	93.1	94.8	95.9	95.8	95.8	95.9	96.0	78.1

As can be seen in Table 1, the adsorption was well-done in the range of pH from 4.0 to 10.0 because the fluoride adsorption efficiencies of RHA/$Al(OH)_3$ were always more than 90% and unvaried, especially, from 6.0 to 10.0. Also, both of studies of Salifu [10] and Ganvir [1] expressed that the pH of 7.0 (± 0.2) had been the suitable pH for fluoride removal. Therefore, the adjustment of pH was no need in after experiments.

3.2. Effect of adsorbent dose

Table 2 shows fluoride removal capacity of RHA/$Al(OH)_3$ with different doses at pH of 7.0. Table 2, it has been found that the fluoride adsorption efficiency increases from 76.7 % to nearly

100 % correspoding with the rise of adsorbent dose from 1 to 5 $g.L^{-1}$. It can be explained that the adsorption surface increases when the dose of material increases. From the dose of 5 to 10 $g.L^{-1}$, almost of fluoride was removed from the solution, so, the adsorption capacity varied unimportant. Hence, 5 $g.L^{-1}$ of RHA/$Al(OH)_3$ was chosen as the optimum dose.

Table 2. The influence of RHA/$Al(OH)_3$ dose on fluoride removal capacity.

Notations	LL1	LL2	LL3	LL4	LL5	LL6	LL7	LL8	LL9	LL10
Dose of adsorbent ($g.L^{-1}$)	1	2	3	4	5	6	7	8	9	10
C_e ($mg.L^{-1}$)	2.33	1.42	0.71	0.56	0.41	0.32	0.25	0.23	0.17	0.15
H (%)	76.7	85.8	92.9	94.4	95.9	96.8	97.5	97.7	98.3	98.5

3.3. Adsorption isotherm study

The correlation between equilibrium concentration of fluoride (C_e) and adsorption amount of RHA/$Al(OH)_3$ (q_e) was considered through isothermal adsorption study. The data acquired from experiments of 7 samples from C5 to C35 with different initial concentrations of fluoride were shown in Table 3. After that, the interaction between q_e and C_e was obtained from Langmuir 2 and Freundlich models. This result was expressed in Figure 1.

Table 3. The fluoride removal capacities at different initial concentrations of fluoride.

Notations	C5	C10	C15	C20	C25	C30	C35
C_o ($mg.L^{-1}$)	5	10	15	20	25	30	35
C_e ($mg.L^{-1}$)	0.222	0.413	0.751	1.278	1.954	2.929	4.449
q_e ($mg.g^{-1}$)	0.956	1.917	2.850	3.744	4.609	5.414	6.110

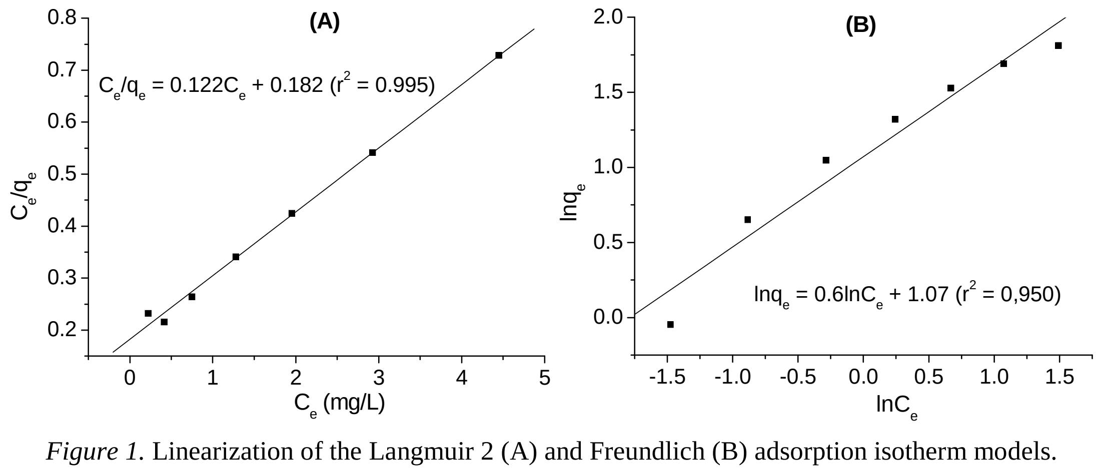

Figure 1. Linearization of the Langmuir 2 (A) and Freundlich (B) adsorption isotherm models.

As can seen, the correlation coefficient (r) value of Langmuir 2 model (r^2 = 0.995) was higher than that of Freundlich model (r^2 = 0.950), which implied that the isotherm data were fitted to the Langmuir 2 model. Maximum fluoride adsorption capacity of material was 8.2

mg.g^{-1}. These results were similar to those in the study of Salifu [10]. Salifu indicated that among adsoprtion isotherm models, Langmuir 2 model exposed the highest correlation coefficient (r^2 = 0.985) with the maximum fluoride adsorption capacity of 7.874 mg.g^{-1}. The results of Z. Qiusheng also proved the suitability of Langmuir 2 model with r^2 = 0.968 [11]. According to this model, the fluoride adsorption onto RHA/$Al(OH)_3$ is monolayer adsorption, that means the surface containing the adsorbing sites is perfectly flat plane with no corrugation, all sites are equivalent, each site can hold at most one molecule of adsorbate and there are no interactions between adsorbate molecules on adjacent sites.

3.4. Adsorption kinetic

In order to find the kinetic model describing well the adsortion of fluoride onto RHA/$Al(OH)_3$, the effect of time to the adsorption was analysed. The amount of fluoride adsorbed onto the material (q_t) at different time (t) was determined. The results were showed in Table 4. The correlation bewteen q_t and t was obtained from these results in Figure 2.

Table 4. The amount of fluoride adsorbed onto the material at different time.

Notations	D10	D20	D30	D40	D50	D60	D70	D80
t (mintues)	10	20	30	40	50	60	70	80
C_t (mg.L^{-1})	1.97	1.64	1.34	1.08	0.86	0.68	0.54	0.42
q_t (mg.g^{-1})	1.606	1.672	1.732	1.784	1.828	1.864	1.892	1.916
Notations	D90	D100	D110	D120	D130	D140	D150	D160
t (mintues)	90	100	110	120	130	140	150	160
C_t (mg.L^{-1})	0.43	0.43	0.42	0.42	0.40	0.43	0.40	0.41
q_t (mg.g^{-1})	1.915	1.914	1.917	1.916	1.92	1.914	1.92	1.918

Figure 2 showed that in the first 80 minutes, the longer time, the more fluoride adsorbed onto the material (q_t). After that, the value of q_t varied around 1.91 mg.g^{-1} which proved that the adsorption got equilibrium.

From the reaction kinetic of pseudo-first-order and pseudo-second-order rate model, the correlation between $\ln(q_e-q_t)$ and t; t/q_t and t was expressed in Figure 3.

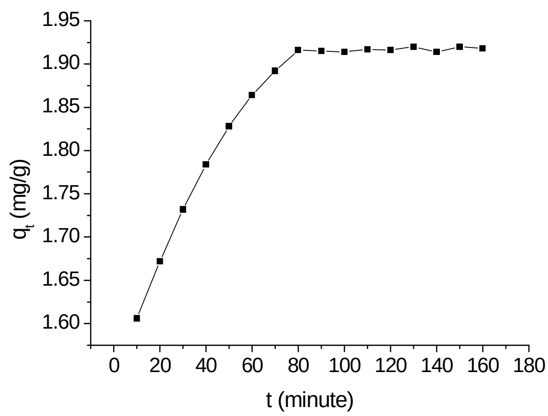

Figure 2. The relation bewteen q_t and t.

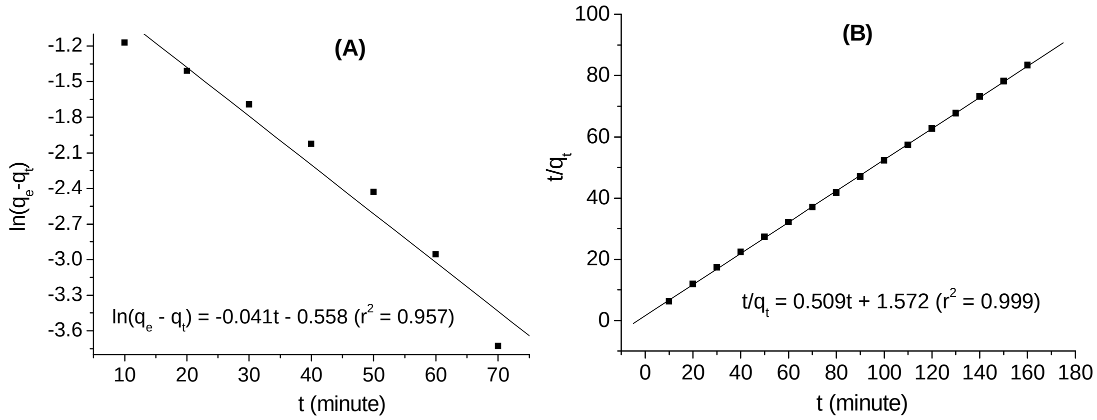

Figure 3. Pseudo-first-order model (A) and pseudo-second-order model (B) for fluoride adsorption.

The above results indicated that the adsorption of fluoride onto RHA/$Al(OH)_3$ obeyed the pseudo-second-order rate model with high correlation coefficient (r^2 = 0.999). This agrees with the publication of Ganvir [1], Garcia-Sanchez [7] and Qiusheng [11].

3.5. Thermodynamic studies

Thermodynamic parameters of fluoride adsorption onto RHA/$Al(OH)_3$ were determined by the investigation of the effect of temperature to the amount of adsorbed fluoride in the range of temperature from 303 K to 343 K. The results were represented in Table 5.

From the Figure 4, thermodynamic parameters were calculated and showed in Table 6.

Table 5. The effect of temperature to fluoride adsorption of RHA/$Al(OH)_3$.

C_0 (mg.L^{-1})	C_e (mg.L^{-1})				
	303 (K)	313 (K)	323 (K)	333 (K)	343 (K)
10	0.43	0.41	0.39	0.36	0.36
15	0.82	0.68	0.63	0.61	0.60
20	1.29	1.18	1.12	1.09	1.06
25	2.15	1.94	1.82	1.69	1.60
30	3.02	2.96	2.87	2.51	2.17
35	4.50	4.27	4.02	3.90	3.88
40	6.55	5.72	5.43	5.01	4.90
q_m (mg.g^{-1})	8.2	8.4	8.5	8.6	8.7
$\ln K_0$	1.672	1.754	1.817	1.868	1.911

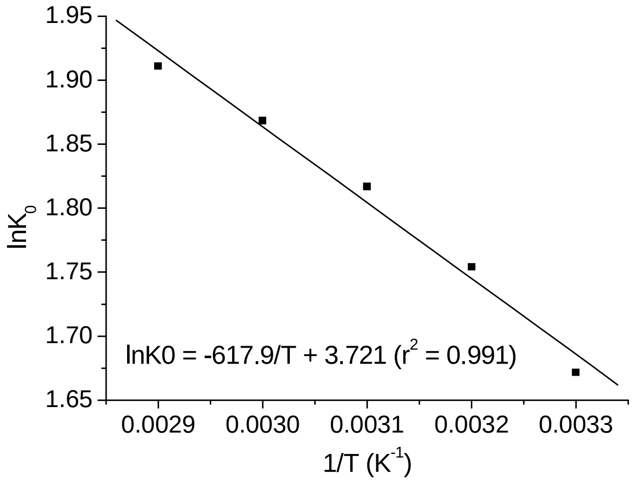

Figure 4. Van't Hoff diagram of fluoride adsorption onto RHA/$Al(OH)_3$.

Table 6. Thermodynamic parameters of fluoride adsorption onto RHA/$Al(OH)_3$.

Temperature (K)	ΔG^o ($J.mol^{-1}$)	ΔH^o ($J.mol^{-1}$)
303	-4211.53	5137.22
313	-4565.09	
323	-4879.69	
333	-5171.87	
343	-5449.63	

The positive value of enthalpy (ΔH^o = +5.14 $kJ.mol^{-1}$) proved endothermic nature of fluoride adsorption, which agreed with the result that the higher temperature, the higher adsorption capacity at equilibrium state, absolutely. The chemisorption nature of the adsorption was also affirmed because of high enthalpy. The mechanism of the adsorption might be ion exchange between fluoride ion and OH^- groups from ≡Al-OH sites [11]. In addition, the higher pH, the higher amount of adsorbed fluoride because the pH of the solution was higher than 5.53 [6], the point of zero charge of material. So, an amount of fluoride was complexed strongly with Al^{3+} on the surface of rice husk ash. The negative value of ΔG^o at different temperatures implied that the adsorption was spontaneous.

3.6. Treatment of fluoride in well-water

In Khanhhoa province, the fluorosis is so popular because of high concentration of fluoride that exceeds the the acceptable limit [2]. For practical purpose, we sampled well-water at Ninhthuong commune, Ninhhoa district, Khanhhoa province. The samples that are transparent, colorless, odorless contain fluoride with the concentration of about 10.1 $mg.L^{-1}$ (exceeds the standard of WHO) and have pH of 8.8.

Ten samples notated from H1 to H10 were prepared. Each sample contained 100 mL of well-water. RHA/$Al(OH)_3$ was added into the sample with the dose increasing from 1 $g.L^{-1}$ to 10 $g.L^{-1}$. The samples were stirred for 2.0 hours in order to reach to adsorption equilibrium. The concentration of fluoride after adsorption were determined (C_e). The results were showed in Table 7.

Table 7. Fluoride adsorption efficiency of $RHA/Al(OH)_3$ in well-water.

Notations	Dose of material ($g.L^{-1}$)	C_e (mg.L)	H (%)
H1	1	3.48	65.5
H2	2	2.40	76.2
H3	3	1.72	83.0
H4	4	1.13	88.8
H5	5	0.76	92.5
H6	6	0.63	93.8
H7	7	0.57	94.4
H8	8	0.49	95.1
H9	9	0.45	95.5
H10	10	0.31	96.9

The above results indicate that the fluoride adsorption ability of $RHA/Al(OH)_3$ in well-water is well. With the dose of material from 4.0 $g.L^{-1}$ to 7.0 $g.L^{-1}$, the fluoride concentration decreases under the acceptable limit of WHO (0.5 - 1.5 $g.L^{-1}$) that is useful for the development of teeth and bond. Well-water after treatment is colorless, odorless, has pH of 7.65 and can be used as running water.

4. CONCLUSION

$RHA/Al(OH)_3$ containing 20 % Al_2O_3 could adsorbed fluoride ion in aqueous solution with the maximum adsorption capacity of 8.2 $mg.g^{-1}$. The adsorption capacity was depended on the solution pH and the adsorbent dose. The adsorption data could be well-described by Langmuir 2 model and the adsorption kinetic followed the pseudo-second-order model. The positive enthalpy (ΔH^o = +5.14 kJ/mol) proved that the chemisorption predominated. This material could well adsorb fluoride in well-water that contained high content of fluoride. After treatment with the dose of material of 4.0 - 7.0 $g.L^{-1}$ for 2 hours, the concentration of fluoride was decreased from around 10.1 $mg.L^{-1}$ to 0.5 - 1.5 $g.L^{-1}$ that was fit for the acceptable limit of WHO.

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TÓM TẮT

NGHIÊN CỨU ĐỘNG HỌC VÀ NHIỆT ĐỘNG CỦA QUÁ TRÌNH HẤP PHỤ ION FLORUA TRONG DUNG DỊCH NƯỚC LÊN VẬT LIỆU TRO TRẤU PHỦ NHÔM HIDROXIT

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Khả năng hấp phụ ion florua trong dung dịch nước bằng vật liệu tro trấu phủ nhôm hydroxit (RHA/$Al(OH)_3$) đã được khảo sát trong nghiên cứu này. Kết quả nghiên cứu đẳng nhiệt cho thấy mô hình đẳng nhiệt hấp phụ Langmuir mô tả tốt bản chất của quá trình hấp phụ với dung lượng hấp phụ cực đại đạt 8,2 mg/g. Đây là quá trình hấp phụ hóa học, tốc độ hấp phụ tuân theo phương trình động học hấp phụ bậc hai biểu kiến. Khảo sát ảnh hưởng của nhiệt độ đến dung lượng hấp phụ trong khoảng nhiệt độ từ 30 đến 70 ^{o}C cho thấy quá trình hấp phụ là tự diễn biến và thu nhiệt. Vật liệu RHA/$Al(OH)_3$ có khả năng xử lý rất tốt các mẫu nước giếng trong thực tế có nồng độ ion florua cao. Với các mẫu nước giếng tại huyện Ninh Hòa (Khánh Hòa) có nồng độ ion florua 10,1 mg/L, sau khi xử lí với liều lượng vật liệu hấp phụ từ 4,0 đến 7,0 g/L, sau thời gian 2 giờ, nồng độ ion florua trong nước giếng sau khi xử lí đạt từ 0,5 – 1,5 g/L, thoả mãn tiêu chuẩn của WHO.

Từ khóa: vật liệu tro trấu phủ nhôm hidroxit, hấp phụ, florua.