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REUSING SLUDGE FROM SURFACE WATER TREATMENT PLANT FOR HEXAVALENT CHROMIUM ADSORPTION IN AQUEOUS SOLUTION

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Abstract. Contaminants in plating wastewater are hard to remove, especially hexavalent chromium ions. This waste stream contains a large amount of Cr^{6+}, a highly toxic metal, potential to cause cancer. To reduce this pollution, sludge from a supply water treatment plant (DWTS) has been used to make adsorbent. This sludge was denaturized by thermal method. After that, a plating synthetic waste stream containing Cr^{6+} was treated using a batch adsorption model with adsorbent made from DWTS. pH, amount of adsorbent, and treatment time were investigated to determine the suitable treatment conditions. The study also evaluated the suitability of two adsorption models, Langmuir and Freundlich. The results showed that, under the conditions of pH = 6.0, the adsorbent content of 25.0 g/L, after 90.0 minutes, the remaining Cr^{6+} content was 0.14 mg/L, close to QCVN 40:2011/BTNMT, column B. The parameters of the Langmuir and Freundlich adsorption equations are: q_{max} = 12.578 mg/g; K_{ads} = 0.747 L/mg; n = 0.338; k_f = 5.138. The chromium adsorption process fits the Freundlich isothermal model better than the Langmuir isothermal model.

Keywords: adsorption, hexavalent chromium, plating wastewater, surface water treatment sludge.

Classification numbers: 3.3.2, 3.4.2, 3.7.3

1. INTRODUCTION

According to Viet Nam's regulations, sludge is stored, collected, and transported to a centralized treatment site. The treatment and reuse of sludge must comply with regulations on management and use of sludge issued by competent Government Agencies, and regulations on environmental protection. In addition, the government prohibits the exchange, sale, and discharge of untreated sewage sludge. If DWTS is determined to contain no impurities, odors, or potential risk of causing environmental pollution by the appropriate authorities, it will be managed similarly to mud. Many standards regulate the discharge of sludge into the soil environment. These standards specify the amount of heavy metals, pollutant content, and limited concentration of pesticides. Some regulations on sludge discharge applied at present are (i) National Technical Regulation QCVN 03:2008/BTNMT (the limit of heavy metals in soil); (ii) The Law on Environmental Protection 2005. If a water treatment plant discharges into the sewer

system, the concentration of pollutants must be lower than the levels specified in QCVN 40:2011/BTNMT (permissible values of pollution parameters in wastewater), and TCVN 6772:2000 (Freshwater quality for aquatic life protection). The problems related to DWTS disposal have not been noticed in Viet Nam for the following reasons: (i) The government and society have not paid attention to this waste as it does not affect significantly on health and environment; (ii) The law on quality assessment and regulations of DWTS discharge is unclear and unification; (iii) The selling price of supply water is still very low, therefore the revenue is not enough to invest in a sludge treatment area.

In supply water treatment technology, DWTS is often generated in the settling and filtration stages. Currently, they are using the following methods to get rid of sludge: (i) Direct discharge into the sewer system; (ii) Recovery of aluminum; (iii) Burial at a landfill; (iv) Some other reuse methods [1]. The method of sludge burial has not been gradually used because of operating costs, shortage of land, as well as the risk of soil and groundwater contamination. The discharge of sludge into rivers or sewers can threaten aquatic lives and damage public construction. Other sludge treatment methods have only achieved the level of research and technology testing. Moreover, these solutions require a reduction in the amount of water in the sludge because the large sludge volume with low solid content will boost the treatment and transportation costs. Therefore, the dewatering process is often applied to cut down tank size and equipment capacity.

Many sludge reusing methods have been proposed and tested such as: composting, cement production, bonsai soil manufacturing, landfill covering material, and ground leveling. The acidification method for aluminum recovery showed an efficiency of more than 80.0 % [2]. However, some toxic metals were also dissolved in this process, and people must eliminate them to prevent secondary pollution. On the other hand, the waste treatment after the recovery process is also complicated and expensive. Fertilization, a mixture of sludge, lime, and some biological substances, is also considered a cheap and straightforward method. This method gives neither real benefit to the soil nor clear economic profit for users compared to the others. Furthermore, it can be easily washed into water resources and makes harm to aquatic lives. Ground leveling and covering were also carried out in some areas. In the rainy season, the land is often muddy, and easily washed away, and in the dry season, dust is easily generated. Weng also developed a method to make bricks and cheap cement from DWTS [3]. Since the elasticity of DWTS is higher than clay, in the calcination process, bricks can easily be cracked and warped. Therefore, this method is only shown in research and demonstration, very difficult to implement in practice.

Besides, hexavalent chromium (Cr^{6+}) from the plating process is toxic to organisms and potentially carcinogenic to humans [4]. Popular traditional technology for this waste stream is coagulation/flocculation which is limited due to the following reasons: (i) large amount of chemicals, (ii) complex operation, and (iii) removal of sludge from the effluent. With the aim of reducing the treatment cost for these wastes, many of them have been reused as disposable adsorbents. In 2011, Trung *et al.* used red mud to adsorb over 90.0 % of Pb^{2+} [5]. In 2016, Thuy *et al.* used DWTS to adsorb 99 % of As^{3+} in raw water [6]. These studies confirm that the adsorbents made from DWTS have many oxides such as Al_2O_3, Fe_2O_3, SiO_2, CaO in large quantities which have the potential to adsorb heavy metals [7]. The application of these adsorbents will reduce sludge loading and wastewater treatment costs.

In the literature, DWTS shows good performance in the adsorption of heavy metals such as Pb^{2+}, Zn^{2+}. However, these ions are easily precipitated at high pH conditions, so it is important to determine the contribution of adsorption or precipitation to the overall treatment efficiency. In this study, we make adsorbents from two types of DWTS. One is from a surface water treatment plant, the other is from a groundwater treatment plant. Then, we compare the adsorption ability

of these adsorbents and analyze the characteristics of the better one. In the next stage, we use this material to adsorb chromium in a synthesized plating wastewater to determine suitable treatment parameters.

2. MATERIALS AND RESEARCH METHODS

DWTS samples are collected from 2 sites: (i) the surface water treatment plant in Hoc Mon District, Ho Chi Minh City; (ii) the groundwater treatment plant in Tan Phu District, Ho Chi Minh City. The sludges were treated by thermal denaturation method according to a previous study [8].

Sludges were cleaned for garbage, dried, pounded for 15 minutes with pestle and mortar. They were completely dewatered by drying at 105 ℃ to constant weight. They were then calcined at specific temperatures (400 ℃, 500 ℃, 600 ℃, 700 ℃). Next, these slurries were finely ground for 60 minutes using an A11 Basic Analytical Mill (IKA - Germany) and sieved through a 0.1 - 0.045 mm sieve. After these steps, we collected two types of materials: surface water sludge adsorbent (SWSA) and groundwater sludge adsorbent (GWSA).

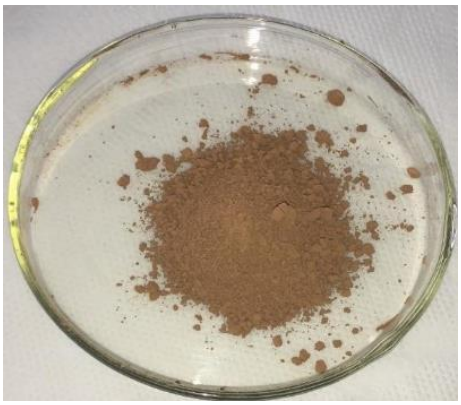

Figure 1. DWTS samples.

Synthetic wastewater is prepared by adding CrO_3 to water to obtain a solution with a Cr^{6+} concentration of 120.0 ± 10.0 mg/L. The adsorption reactor in this experiment consisted of a 250 mL glass reaction flask and a water bath thermostat with magnetic stirrer (NB302N -N-Biotek - Korea) with adjustable speed from 100 to 150 rpm, and a temperature of 25 ℃. 200 mL of wastewater was used for each experiment. pH, amount of adsorbent, and adsorption time were adjusted depending on the requirements of each treatment. The wastewater is filtered through 0.4 µm filter paper to separate the adsorbent at the end of the process and analyzed for Cr^{6+} to evaluate the adsorption capacity.

The XRF method is used to determine the metal oxide composition in SWSA (S2 PUMA - Bruker - Germany), and XRD analysis (S2 RANGER - Bruker - Germany) identifies the crystalline phases present in SWSA and reveals information about the chemical composition. The material morphology is determined by TEM imaging method (JEM 1400 Plus - Jeol - USA) and BET analysis (Autosorb-iQ - Quantachrome Instrument, USA) is used to identify the surface area of materials. pH is directly controlled with a Milwaukee MW 101 Meter handheld pH meter. Cr^{6+}concentration is analyzed by AAS method (Analyst 400 Perkin Elmer, USA).

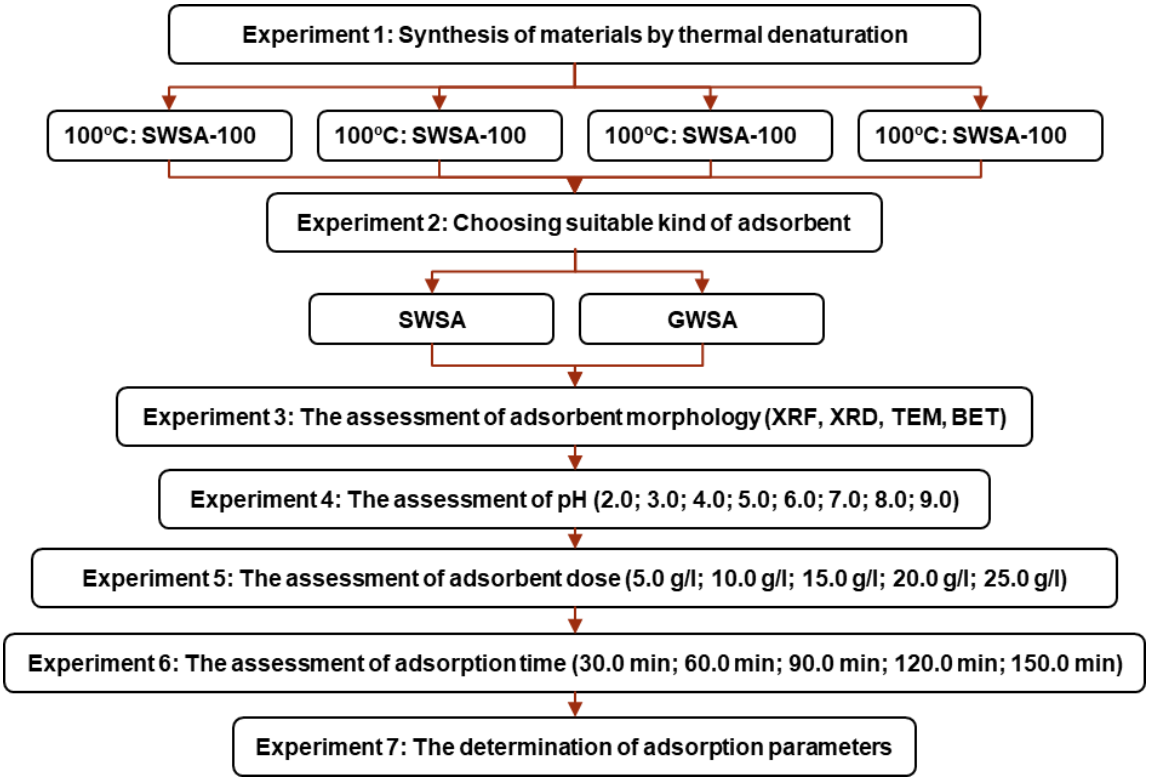

Figure 2. Experimental procedures.

3. RESULTS AND DISCUSSION

3.1. Evaluation of adsorbent materials

Sludge characteristics of water treatment plants in Ho Chi Minh City show that sludge samples from water treatment plants along the Dong Nai River have high concentrations of VDS, pesticides, and heavy metals [1]. This is a very long river, flowing through many areas with diverse domestic and industrial activities. The Saigon River is quite short and there are few industrial zones nearby, so the sludge composition of the water treatment plants near this river contain less organic matter, heavy metals, and pesticides. In addition, sludge from ground water treatment plants was also surveyed and found to have similar composition and low toxic substances, but their composition was quite different from river samples. Since then, this study has selected 2 sludge samples from the water treatment plant at Hoc Mon (using water from the Saigon River) and the water treatment plant at Tan Phu district (using ground water) because they are low in toxic substances and convenient for sampling.

Waste sludge from the surface water treatment process has a high content of Al_2O_3 which is very easy to disperse. Thus, its structure is defective, and the capillary volume and surface area are high. These characteristics are suitable for an adsorbent [9]. The experiments comparing the adsorption capacity of the two kinds of adsorbent at various denaturation temperatures showed that the adsorption efficiency of SWSA is better than GWSA at all denaturation temperatures (Figure 3). Sludge has not been thermally denatured, giving low adsorption efficiency. The evaluation of adsorption capacity showed that SWSA has potential for heavy metal adsorption.

At pH = 8.0, after 60 minutes, the adsorption efficiency is nearly 100 %. This result is higher than the result of Thang *et al.* in 2011 [10]. From this result, we choose SWSA as the suitable adsorbent for the next experiments.

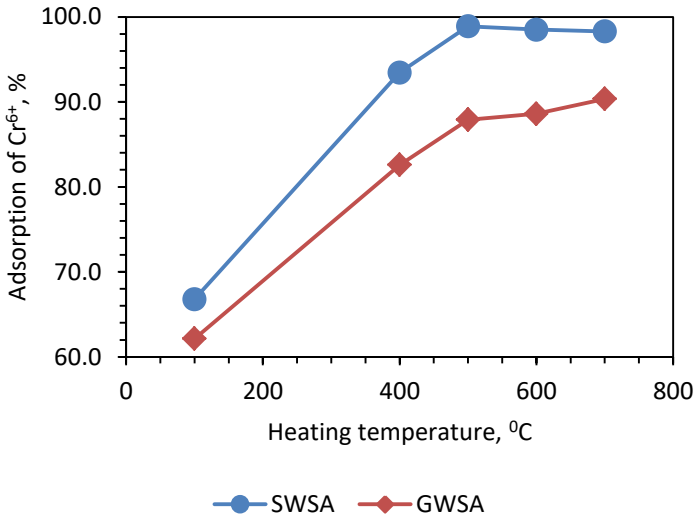

Figure 3. The adsorption of Cr^{6+} of SWSA and GWSA at various denaturation temperatures, pH of 8.0; catalyst dose of 10.0 g/L; treatment time of 60 mins.

The denaturation temperature also dramatically affects the adsorption process. Adsorbent materials calcined at temperatures lower than 500 ºC have not achieved high adsorption efficiency. At the denatured temperatures of 500 ºC, 600 ºC, 700 ºC, the adsorption efficiencies were similar (Figure 3). The reason may be that at temperatures lower than 500 ºC, the organic matter in SWSA has not been decomposed. When they are vaporized, the material forms voids and pores inside the grain structure. This will increase the surface of SWSA particles, leading to an increase in the adsorption capacity [11]. On the other hand, Abo-El-Enein (2017) also explains this phenomenon as follows: (i) the microstructure of SWSA calcined at 100 °C demonstrates a more porous structure consisting primarily of mesoporous. So, it could not get good adsorbability. (ii) At a calcination temperature of 500 ℃, many albite grains are formed, they appear as filaments along with almost amorphous and microcrystalline particles around them. That structure would lead to an increase in the surface area to adsorb metal ions from the aqueous solution. Also, at this temperature the pore system is composed mainly of mesopores which can well adsorb heavy metals. (iii) The albites disappear when heated at 700 ℃. This has resulted in a decrease in surface area and reduced metal adsorption capacity [11]. In this study, we chose SWSA calcined at 500 ºC as a suitable adsorbent with good performance and low synthesis cost.

3.2. Assessment of SWSA morphology

The results in Table 1 show that SWSA-500 contains many oxides, such as CaO, SiO_2, Fe_2O_3, and Al_2O_3. SiO_2 can adsorb heavy metals (Zn^{2+}, Cu^{2+}, Cr^{6+}, etc.) [12]. It is in the form of microcrystalline that agglomerates into blocks of SiO_2 with porous structure. This is an outstanding feature of SiO_2 for adsorption [11]. Besides, the adsorbent also contains a lot of Fe_2O_3 because of the alluvium in the surface water. Fe_2O_3 also has good potential for the

adsorption of heavy metals; they have a spherical shape, uniform particle size (< 10 nm) and cluster together [13]. In addition, PAC (poly aluminum chloride) is also used for coagulation in the surface water treatment plant, so it contains a large amount of Al_2O_3. This oxide is known for the following adsorption characteristics: high dispersion, structural defects, large capillary volume, and extensive surface area [9]. CaO in the sludge has no adsorbability but it can increase the pH of the solution, so it will reduce the amount of pH adjustment chemicals. The other ingredients are in small proportions and have little impact on the environment and human health.

Table 1. XRF analysis of SWSA adsorbent.

No.	Oxide	Percent, %	No.	Oxide	Percent, %
1	MgO	0.46	7	Fe_2O_3	14.74
2	Al_2O_3	28.32	8	SrO	0.00
3	SiO_2	36.86	9	Sb_2O_3	0.00
4	K_2O	0.62	10	BaO	0.00
5	CaO	18.63		**Total**	**100.00**
6	MnO	0.37			

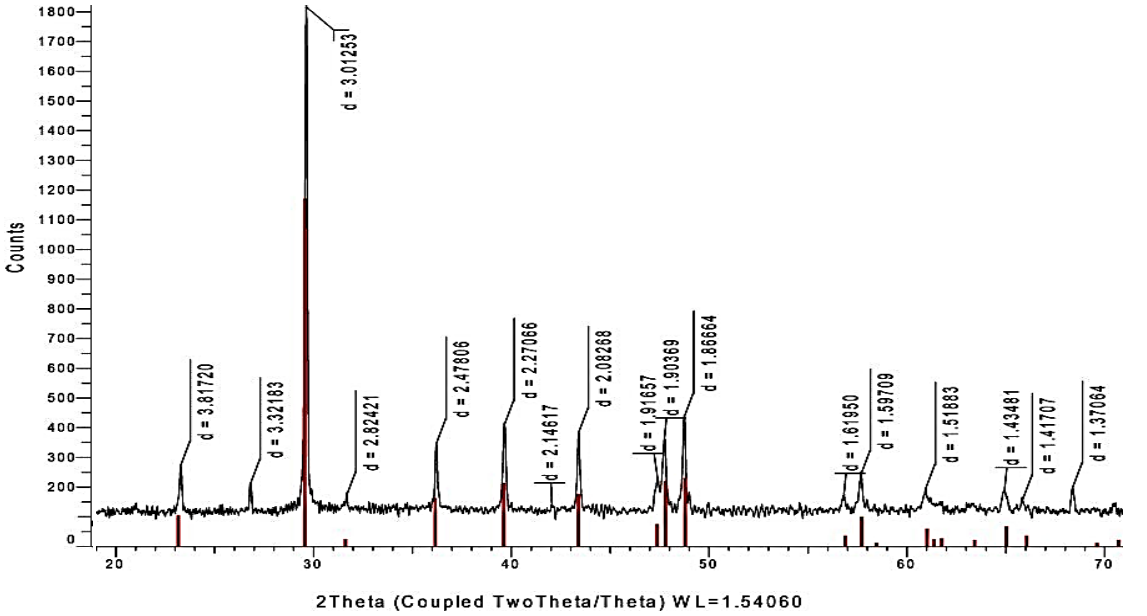

Figure 4. XRD spectrum of SWSA adsorbent at the calcination temperature of 500 ℃.

X-ray diffraction spectrum (Figure 4) appears in the specific pattern of 2θ reflected on the XRD pattern of some oxide crystals as follows: (24^o), (36^o), (43^o) represent for Fe_2O_3 crystal; (27^o), (61^o), (69^o) represent for MgO crystal; (29^o - 30^o) represent for SiO_2 crystal; (36.5^o), (39.5^o), (43.5^o), (48^o) represent for Al_2O_3 crystal and some other impurities. This result is comparable to the study of Hung [14]. Together with the XRF data in Table 1, the sludge composition contains mainly Al_2O_3, SiO_2, and Fe_2O_3.

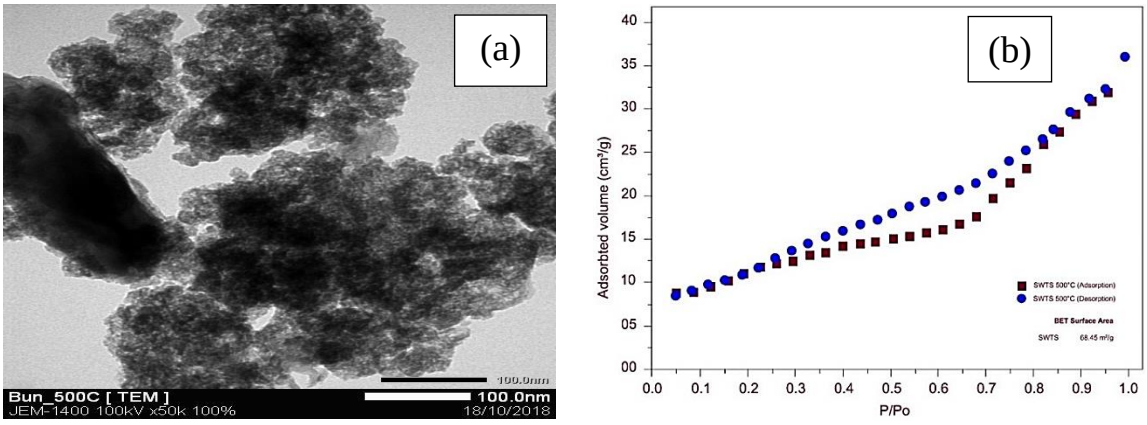

Figure 5. (a) TEM graph and (b) BET plot of SWSA at the calcination temperature of 500 ℃.

Figure 5a shows that the adsorbent is very porous and has many hollow slits, ideal for a suitable adsorbent. This may be due to the evaporation of organic matter from the sludge. On the other hand, the BET plot (Figure 5b) shows that the surface area of the SWSA material is 68.45 m^2/g. Since then, SWSA has got a smaller surface area than activated carbon (200.0 - 300.0 m^2/g) [15] and more extensive than rice husk (22.64 m^2/g) [16].

3.3. Assessment of the effect of pH on the adsorption of Cr^{6+}

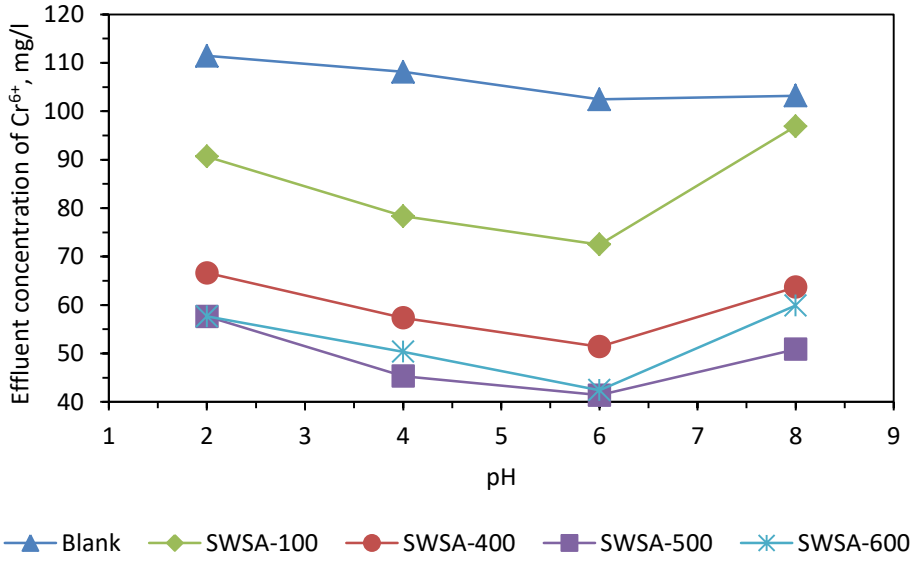

Figure 6. The influence of pH on the adsorption of Cr^{6+} under the following conditions: initial Cr^{6+} concentration of 120.0 mg/L; SWSA dose of 1.0 g/L, treatment time of 60 mins.

* *Note:* blank: experiment without adsorbent; SWSA-100, SWSA-400, SWSA-500, and SWSA-600 are SWSA calcined at 100 ℃, 400℃, 500 ℃, and 600 ℃, respectively.

The experimental results (Figure 6) show that when the pH value is adjusted from 2.0 to 6.0, the Cr^{6+} adsorption efficiency of the materials increases. When the pH value is higher than 6.0, the adsorption efficiency decreases for all adsorbents. SWSA-500 shows the best

performance, where the concentration of Cr^{6+} after treatment decreased from pH = 2.0 (57.65 mg/L) to pH = 6.0 (42.41 mg/L). It can be explained as follows: in a strongly acidic aqueous solution, both adsorbent and adsorbate particles are positively charged, so the interaction force is electrostatic repulsion. Furthermore, when the pH value is low, the concentration of H^+ in the solution is huge. It will compete with metal ions during the adsorption process [17]. Similarly, when the pH is high, the concentration of H^+ decreases, while the metal cation concentration is almost constant, so the adsorption of metal ions will be more favorable. In the pH range of 4.0 - 6.0, the active sites on the surface of the adsorbent are protonated and positively charged. Cr^{6+} ions mainly exist as $HCrO_4^-$ anion complex. Therefore, the adsorption process occurs due to the electrostatic affinity between the positively charged adsorbent particles and the negatively charged $HCrO_4^-$ anions [18]. In contrast, the decrease in adsorption efficiency at high pH (pH > 6.0) is because of the competition of Cr^{6+} and OH^- ions in water [11]. The experiment is also performed with a blank sample (without SWSA), but the removal of Cr^{6+} ions is not significant. The adsorption capacity of the materials also increased gradually with the calcination temperatures in the order: SWSA-100 < SWSA-400 < SWSA-500. They become similar at the calcination temperature of 500 ℃ and 600 ℃. The pH value of 6.0 is considered suitable for the treatment and it is used for further experiments.

3.4. Assessment of adsorbent dose

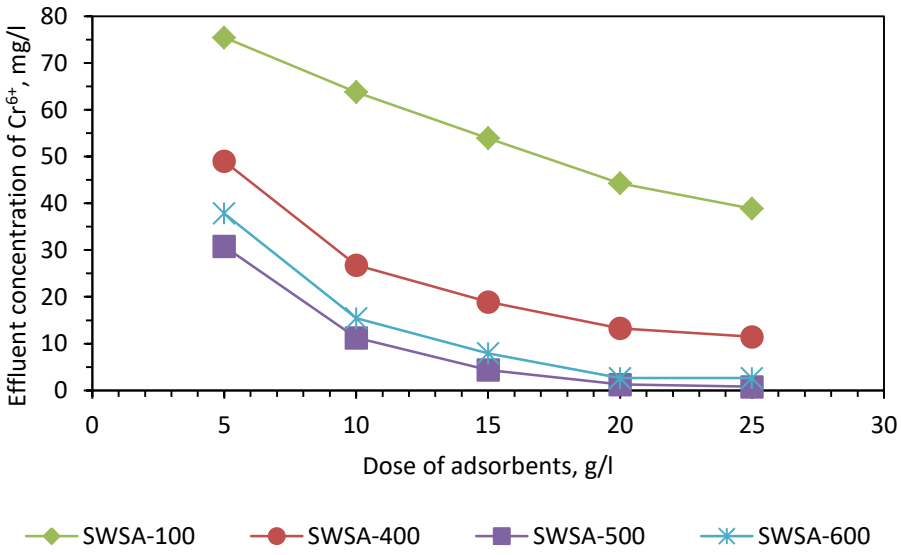

Figure 7. The influence of adsorbent dose on the adsorption of Cr^{6+} under the following conditions: initial Cr^{6+}concentration of 120.0 mg/L; pH of 6.0; treatment time of 60 mins.

The results in Figure 7 show that when increasing the adsorbent dose, the amount of Cr^{6+} decreases gradually. The lowest Cr^{6+}concentration after treatment was 0.78 mg/L, corresponding to the adsorbent dose of 25.0 g/L. However, the Cr^{6+}content has not yet reached the Vietnam national technical regulation on industrial wastewater QCVN 40:2011/BTNMT. This can be explained as follows: When the dose of SWSA increases, the adsorption also improves because metal ions attach to functional groups in the adsorbent and cause the separation of H^+ ions [11]. If SWSA was used in high quantity, it could adsorb a small amount of Cr^{6+} ions. This can become a challenge for the sludge removal after the adsorption. The calcination temperature of

500 ℃ and 600 ℃ gives similar treatment efficiency in the catalyst dosage range of 20.0 and 25.0 g/L. SWSA-100 and SWSA-400 show poor performance, while the adsorption capacity of SWSA-500 and SWSA-600 is excellent and comparable. The appropriate dose of SWSA-500 in this case is 25.0 g/L.

3.5. Assessment of adsorption time

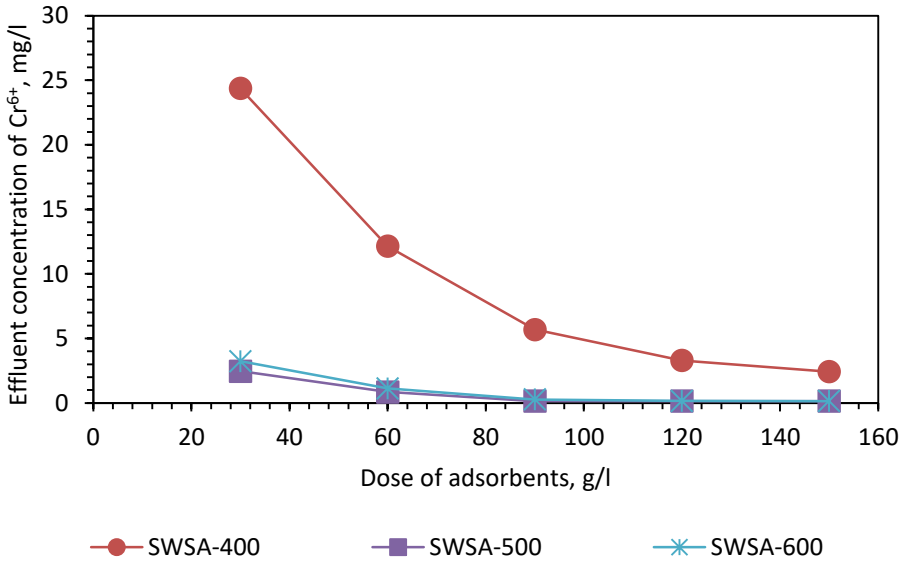

Figure 8. The influence of treatment time on the adsorption of Cr^{6+} ion under the following conditions: initial Cr^{6+} concentration of 120.0 mg/L; pH of 6.0; SWSA dose of 25.0 g/L; calcination temperature of 500 ℃.

The results in Figure 8 show that when the adsorption time is prolonged, the treatment efficiency also grows up. SWSA-500 and SWSA-600 materials have better adsorption capacity than SWSA-400. After 90 minutes with SWSA-500 material, the remaining amount of Cr^{6+} is 0.14 mg/L and changes slightly later. Since adsorption is a reversible process, it needs to stop the process to avoid desorption when the adsorption process stabilizes. Regarding the contact time between the adsorbent and the adsorbate, when the treatment time is short, it is not enough for the active sites on the surface of the adsorbent to be filled with Cr^{6+}. On the contrary, when the adsorption time is extended, the amount of adsorbent accumulated on the surface of the adsorbent is also high. The rate of desorption of the adsorbate into the water also increases. At this time, the adsorption efficiency will not increase and gradually reach the equilibrium state [19]. Therefore, the contact time of 90 min is considered as the suitable adsorption time. This result is higher than the studies of Minh and Bulut (60 mins) [20, 21], but lower than the studies of Özer (120 mins) [22], and Garg (240 mins) [23].

3.6. Determination of adsorption parameters according to Langmuir and Freundlich isotherm models

Langmuir and Freundlich isotherm models are two popular models used to describe the adsorption process. In particular, the Langmuir isotherm equation describes the adsorption process on a homogeneous adsorption surface. It is widely used to process adsorption data and

calculate the maximum adsorption capacity in adsorption processes [24]. The Freundlich model shows an empirical equation for adsorption on heterogeneous surfaces [24]. To determine the required adsorption parameters for SWSA materials, two models, Langmuir (1) and Freundlich (3), are linearized and shown in equation (2) and equation (4) [25].

$$q = \frac{q_{max} K_{ads} c}{1 + K_{ads} c} \quad (1)$$

$$\frac{1}{q} = \frac{1}{q_{max} K_{ads}} \frac{1}{c} + \frac{1}{q_{max}} \quad (2)$$

$$q = k_f c^n \quad (3)$$

$$\ln q = \ln k_f + n \ln c \quad (4)$$

where: q is the adsorption capacity at the equilibrium time of the adsorbent; n is the constant indicating the adsorption strength, K_f is the Freundlich equilibrium constant, and K_{ads} is the Langmuir equilibrium constant; q_{max} is the maximum adsorption capacity (mg/g); C is the concentration of the adsorbent at the required time.

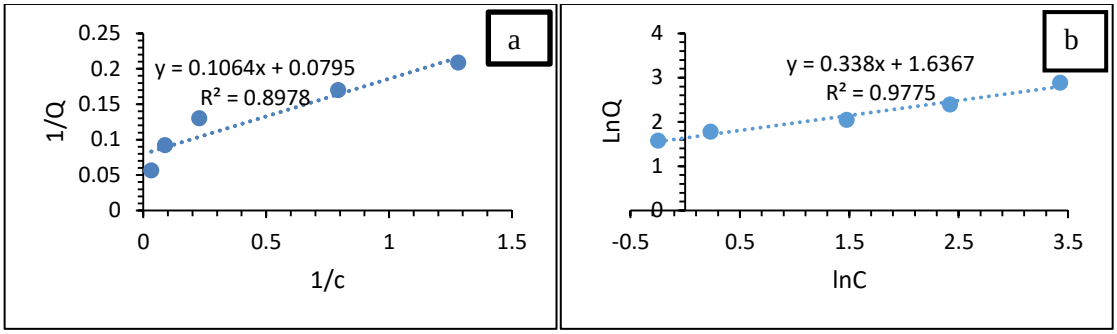

Figure 9. (a) Langmuir and (b) Freundlich isotherm models.

The experimental results are shown in Figure 9. Using the least squares method, the Langmuir and Freundlich isotherm equations are determined as equation (5) and equation (6), respectively.

$$\frac{1}{q} = 0.1064 \frac{1}{C} + 0.0795 \quad (5)$$

$$lnq = 0.338 lnC + 1.6367 \quad (6)$$

$$R_L = \frac{1}{1 + K_{ads} c_o} \quad (7)$$

from the regression equations (5) and (6), the parameters of the adsorption equation are determined: q_{max} = 12.578 mg/g; K_{ads} = 0.747 L/mg; n = 0.338; k_f = 5.138. The maximum Cr^{6+}adsorption capacity of SWSA-500 was higher than that of Cr^{6+}adsorption with red mud (2.34 mg/g) [20], winery sludge (5.7 mg/g) [26], and lower than activated charcoal (18.591 mg/g) [27]. K_{ads} is in the range of 0 - 1, indicating that the adsorption process is consistent with the Langmuir model. The R_L value shows the adsorption preference; the calculation result of the R_L value (7) is 0.011 (< 1.000), showing that the adsorption process of Cr^{6+} by SWSA-500 is very favorable and reversible [25]. The n value in the Freundlich equation is used to evaluate whether the adsorption process is either physical (n > 1.000) or chemical (n < 1.000). Thereby, the adsorption process of Cr^{6+} by SWSA-500 with n = 0.338 shows that chemical adsorption is

dominant over physical adsorption and Cr^{6+} is adsorbed favorably on the surface of the adsorbent [25]. This conclusion is applicable with the nature of this study because the purpose of thermal modification is to decompose the organic matter in the sludge to increase the surface area, thereby enhancing the adsorbability. The coefficient of determination (R^2) for equations (5) and (6) are 0.8978 and 0.9775, respectively. This shows that the experimental data have high reliability, and the adsorption process of SWSA-500 materials has a better fitting with the Freundlich isotherm model than the Langmuir isotherm model.

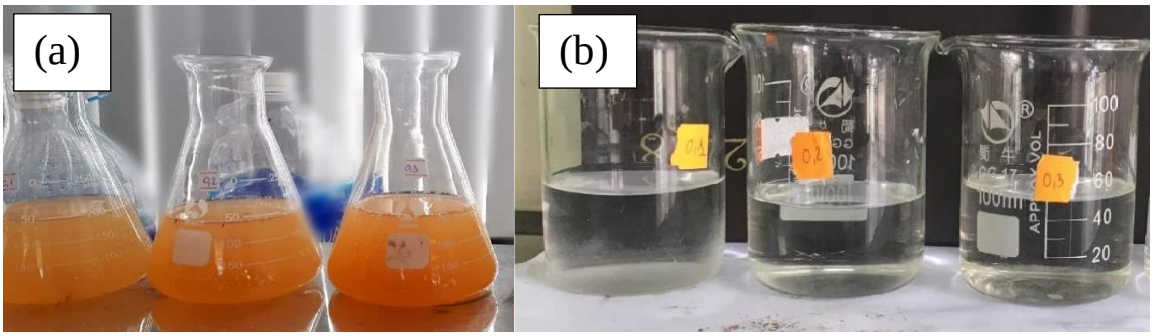

Figure 10. (a) Experiment samples on treatment; (b) Water samples after treatment.

4. CONCLUSIONS

The adsorbent material made from surface water treatment process sludge has a good Cr^{6+}adsorption capacity. Under the following conditions: calcination temperature of 500 ℃, pH of 6.0, sludge dosage of 25.0 g/L, contact time of 90 mins, the Cr^{6+}content in the effluent is 0.14 mg/L, close to the Vietnam national technical regulation on industrial wastewater QCVN 40: 2011/BTNMT, column B (0.10 mg/L). The parameters of the Langmuir and Freundlich adsorption isotherm equations are: q_{max} = 12.578 mg/g; K_{ads} = 0.747 L/mg; n = 0.338; k_f = 5.138. The Cr^{6+}adsorption process by SWSA-500 is more consistent with the Freundlich isotherm model than the Langmuir isotherm model.

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Authorship contribution statement. Author 1: Methodology, Investigation, Funding acquisition. Author 2: Formal analysis. Author 3: Formal analysis, Supervision. etc.

Declaration of competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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