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To cite this article: Mutairah Shaker Alshammari, I. M. Ahmed, Jaber S. Alsharari, Ibrahim Hotan Alsohaimi, Nayef S. Al-Muaikel, Thamer S. Alraddadi & Tamer H. A. Hasanin (2021): Adsorption of Cr(VI) using α -Fe₂O₃ coated hydroxy magnesium silicate (HMS): isotherm, thermodynamic and kinetic study, International Journal of Environmental Analytical Chemistry, DOI: [10.1080/03067319.2021.1890061](https://doi.org/10.1080/03067319.2021.1890061)

To link to this article: <https://doi.org/10.1080/03067319.2021.1890061>



Published online: 01 Mar 2021.



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Adsorption of Cr(VI) using α -Fe₂O₃ coated hydroxy magnesium silicate (HMS): isotherm, thermodynamic and kinetic study

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ABSTRACT

α -Fe₂O₃ coated hydroxy magnesium silicate (HMS) was prepared by the coprecipitation method and was used for the sorption of Cr(VI) from the aquatic environment. FT-IR, XRD, and SEM characterised the investigated composite. The influence of pH, shaking time, Cr(VI) concentration, and the temperature was investigated. The results obtained are fitted well with the pseudo-second-order kinetic model. Besides, Freundlich isotherm is adequate than the Langmuir isotherm. The adsorption was observed to be exothermic, spontaneous, and accompanied by increasing the system's randomness. The adsorption of chromium attained equilibrium after 60.0 min. The maximum adsorption capacities of α -Fe₂O₃/HMS composite for Cr(VI) were 10.75 mg/g obtained at a pH of 2.

ARTICLE HISTORY

Received 5 December 2020
Accepted 27 January 2021

KEYWORDS

Cr(VI); sorption; α -Fe₂O₃/HMS composite; exothermic

1. Introduction

Hydroxyl magnesium silicate (HMS), talc powder, is used in many applications as additives in paints, organic agriculture, clarifier in food industry, cosmetics, fire retardant and wastewater treatment [1–3]. The chemical formula of HMS is Mg₃Si₄O₁₀(OH)₂ that consists of SiO₂ (62.70 wt.%), MgO (28.50 wt.%), CaO (4.88 wt.%), H₂O (4.76 wt.%) in addition to other oxides [4]. The brucite octahedral magnesium hydroxide structures sandwiched between two tetrahedral silica sheets through ionic and covalent bonds [5]. Figure 1 shows the schematic structure of HMS [6]. HMS finds wide applications as a good sorbent for metal ions, dyes [7–11], and radioactive waste encapsulation [12–19]. Many modifications were proposed on the surface of HMS in order to improve its sorption capacity and enhances its physicomechanical properties [20–22].

Chromium present in many oxidation states varies from +2 to +6, while the trivalent and hexavalent are of environmental significance. In natural waters, only Cr(III) and Cr(VI) exist. The existence and ratio between these two oxidation states are affected by many parameters as the chemical and photochemical redox, adsorption-desorption,

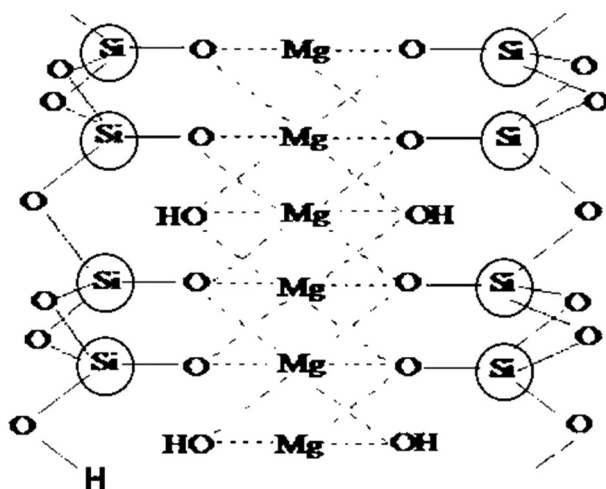


Figure 1. Schematic representation of HMS.

precipitation-dissolution reactions, quantity of organic matter and dissolved oxygen [23]. Hexavalent chromium is found in the effluents of many industries as leather tanning, ferrochrome, colouring glass, wood preservation, metal finishing, electroplating, pigment manufacture, combustion of fossil fuel and others that are highly toxic owing to its mutagenic and carcinogenic characteristics [24]. Cr(VI) is also found in intermediate and low-level active wastes as it considered a corrosion product for the stainless steel nuclear reactors. Cr(VI) in water has a permissible limit of 0.05 ppm according to the World Health Organisation (WHO) [25]. Dichromate, $\text{Cr}_2\text{O}_7^{2-}$ and bichromate, HCrO_4^- ions are highly mobile in soils thus can be absorbed by plants and leached out into the ground and surface water causing water pollution [26] while Cr(III) is primary micro-nutrient for the glucose and lipid metabolism in mammals [27]. Adsorption is one of the more popular methods. Many adsorbents were investigated, such as clay minerals from which, Zeolite [28], bentonite [29,30], graphene nanosheets [31], functionalised graphene oxide [32], modified montmorillonite [33], mesoporous silica [34], dolomite [35], Layered double hydroxide, LDH [36,37], kaolinite [38], Natural Goethite [39] and bio sorbents [40–46]. Many authors investigate the sorption of Cr(VI) by iron oxide minerals, such as magnetite, maghemite, haematite, and goethite that find many applications in wastewater treatment [47–50]. To the best of our knowledge, there is not enough study concerning the sorption of chromium(VI) using $\alpha\text{-Fe}_2\text{O}_3$ coated HMS mineral from aqueous solution. In this concern, the synthetic haematite was capped with a silica shell using HMS. This composite has many advantages as the presence of many active that can interact with the chromium species through the hydroxyl groups ($-\text{SiOH}$) and ($-\text{MgOH}$) that cause hydrophilic effect [21], the unique amphoteric properties of iron oxides favour the presence of neutral, positive or negative surface charge of the Fe-oxide that enables the sorption of either anions or cations from aqueous environment [51]. In addition to its simple synthesis in the laboratory, low-cost and high chemical stability gives these composite advantages over other sorbents. In the work described here we studied the sorption and desorption of Cr(VI) six heavy by $\alpha\text{-Fe}_2\text{O}_3$ coated HMS.

2. Experimental

2.1. Chemicals

Analytical grade of $K_2Cr_2O_7$, $FeCl_3 \cdot 6 H_2O$, $FeCl_2 \cdot 4 H_2O$ and NaOH were delivered from Sigma Aldrich while hydroxyl magnesium silicate (HMS) was purchased from local market.

2.2. Preparation of HMS/ α -Fe₂O₃ composite

The HMS/ α -Fe₂O₃ composite was prepared by co-precipitation. First, 2.0 g of HMS was stirred with 100 mL deionised water containing 1.351 g $FeCl_3 \cdot 6 H_2O$ (0.05 M) and 0.497 g $FeCl_2 \cdot 4 H_2O$ (0.025 M), are added to the suspended solids for 2.0 hr at 80°C, the precipitation is attained by dropwise addition of sodium hydroxide (2.0 M), with vigorous stirring till pH 9.5 in nitrogen atmosphere. The magnetite was isolated by a magnet, rinsed with deionised water, then dried and calcined for 2.0 h at 500 °C, then placed in a polyethylene tube for adsorption experiments [21].

2.3. Instrumentation

Perkin Elmer UV–Visible Recording Spectrophotometer was utilised for determination the quantity of Cr(VI). FT-IR spectra of α -Fe₂O₃/HMS composite were performed in a wavelength range of 400–4000 cm^{-1} , using (FTIR)IR – Tracer 100, Shimadzu, Japan. The surface area was characterized utilizing a gas sorption analyser (Quantachrome, NOVA 4200 e series, USA), XRD pattern of the adsorbent was measured using CuK α radiation by Shimadzu X-Ray powder diffractometer (XRD 7000) within 2 θ ranging from 10° to 80°. The morphology of the composite was conducted through scanning electron microscopy (SEM) on Thermo Scientific Quattro ESEM (Thermo Fischer, Waltham, MA, USA).

2.4. Sorption of Chromium (VI) Ions onto α -Fe₂O₃ coated HMS

The batch technique was applied to investigate the sorption studies. Generally, 0.06 g of the composite was shaken with 10 mL of 50 mg/L Cr(VI) ions for 2.0 h at pH 2.0, unless otherwise stated. The quantity of Cr(VI) was estimated by UV-Vis spectrophotometer using 1,5-Diphenylcarbazide method at 540 nm [52]. The uptake % was calculated as the following:

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

where C_o and C_e are the initial and equilibrium concentration of Cr(VI) ions.

3. Results and discussion

3.1. Characterisation of the adsorbents

Figure 2 displays the main diffraction peaks of the composite. HMS shows the diffraction peaks of magnetite ($2\theta = 18.9, 48.6^\circ$), quartz ($2\theta = 28.1^\circ$), dolomite ($2\theta = 33.1^\circ$) while

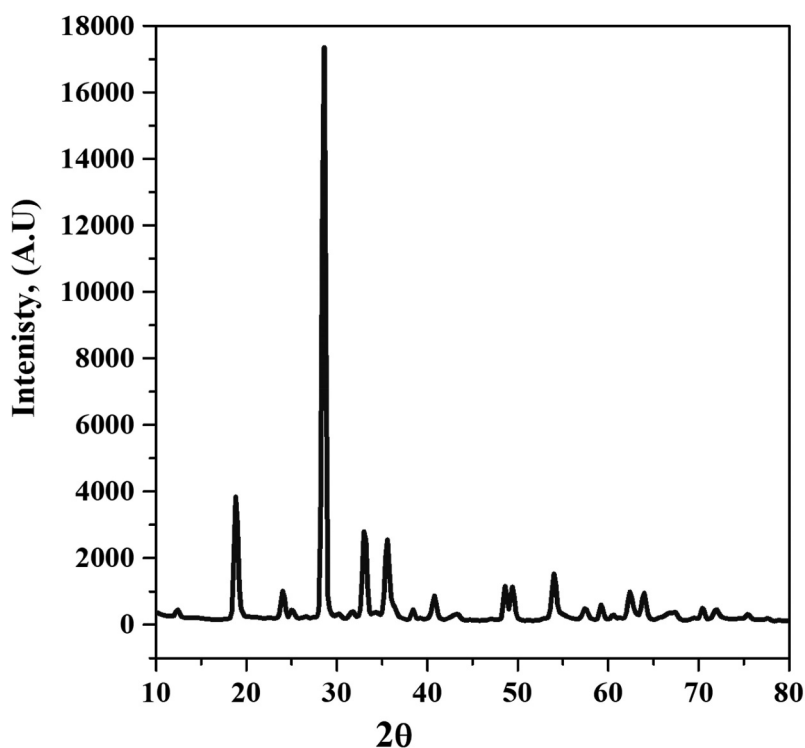


Figure 2. XRD pattern of α -Fe₂O₃/HMS composite.

$2\theta = 24.1, 33.1, 35.6, 40.8, 49.4, 54.0, 62.4$ and 63.9° represents α -Fe₂O₃ of the phases; 012, 104, 110, 113, 024, 116, 214 and 024, respectively, [53–57].

Figure 3 Shows the HMS IR spectrum, a small, sharp band at 3676 cm^{-1} attributed to inner-surface O – H group stretching mode. Bending vibrations mode of Si–O refer to the bands between 400 and 515 cm^{-1} [58,59]. Stretching vibrational bands of siloxane group (Si–O–Si) appear 1018 and 465 cm^{-1} . Mg–O out-of-plane and bending vibration and Si–O–Mg bond appear at 537 and 670 cm^{-1} , respectively. The strong band at $765, 692\text{ cm}^{-1}$ are assigned Fe–O stretching mode, the band at 1100 cm^{-1} refers to silicate [60]. The BET surface area was measured to be $8.45\text{ m}^2/\text{g}$. The SEM of the sample, **Figure 4** shows a plated-like pentagonal particle.

3.2. Effect of pH on the removal of Cr(VI)

The effect of pH on the sorption of 50 mg/L Cr(VI) onto α -Fe₂O₃/HMS composite was investigated in the range (2.0–9.0) at V/m ratio 0.17 Lg^{-1} , **Figure 5**. It is clear that the sorption of Cr(VI) declined dramatically with raising the pH of the aqueous solution. **Figure 6** represents the speciation of Cr(VI) in the pH range 1–12 at ionic strength 0.01 and Cr(VI) concentration of 1.0 mM at ambient temperature that was calculated by MEDUSA programme [61]. Various chromium species exist as $\text{Cr}_2\text{O}_7^{2-}$, HCrO_4^- , CrO_4^{2-} and H_2CrO_4 . The high uptake at lower pH can be justified by an increased number of H⁺ ions, which overcharge the surface of the adsorbent, which exhibited initial negative charge that

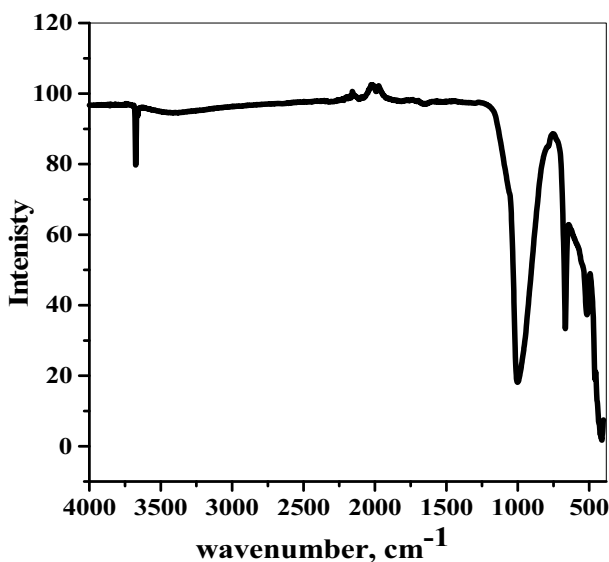
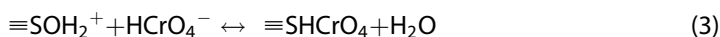
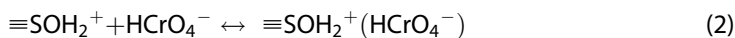


Figure 3. FT-IR of α -Fe₂O₃/HMS composite.

creates electrostatic attraction force between the negatively charged bichromate species (HCrO_4^-), the predominant species at pH 2.0 and the positively charged adsorbent surface in the acidic medium [62]. As the pH increases a repulsion force occurs between the chromate species ($\text{Cr}_2\text{O}_7^{2-}$, CrO_4^{2-} and OH^-) that explained the dramatic decrease in the sorption process. There are two mechanisms may be proposed as:



While $\equiv\text{SOH}_2^+$ represents protonated surface sites. All possible interactions between Cr(VI) and α -Fe₂O₃/HMS composite is illustrated in Figure 7.

3.3. Effect of sorbent dose

The impact of α -Fe₂O₃/HMS dose on the sorption of Cr(VI) ions was examined within the range 0.3–0.12 g/10.0 mL solution of 50 mg/L of Cr(VI) at ambient temperature (25°C) for 60 min. As seen from Figure 8, at low dose, the removal of Cr(VI) increases with raising the sorbent dose owing to increasing the number of active centers accessible for adsorption while at high dose slight increasing was observed and may be due to agglomeration of the adsorbent [63]. In this respect, V/m ratio 0.17gL⁻¹ was chosen for the further investigations.

3.4. Sorption kinetics

The impact of removal time on the uptake of Cr(VI) was examined in the range between 5.0 and 120.0 min. at 25, 35, 45 and 55°C with initial concentration 50.0 mg/L of chromium(VI) onto α -Fe₂O₃/HMS composite, Figure 9, the raising the time of contact,

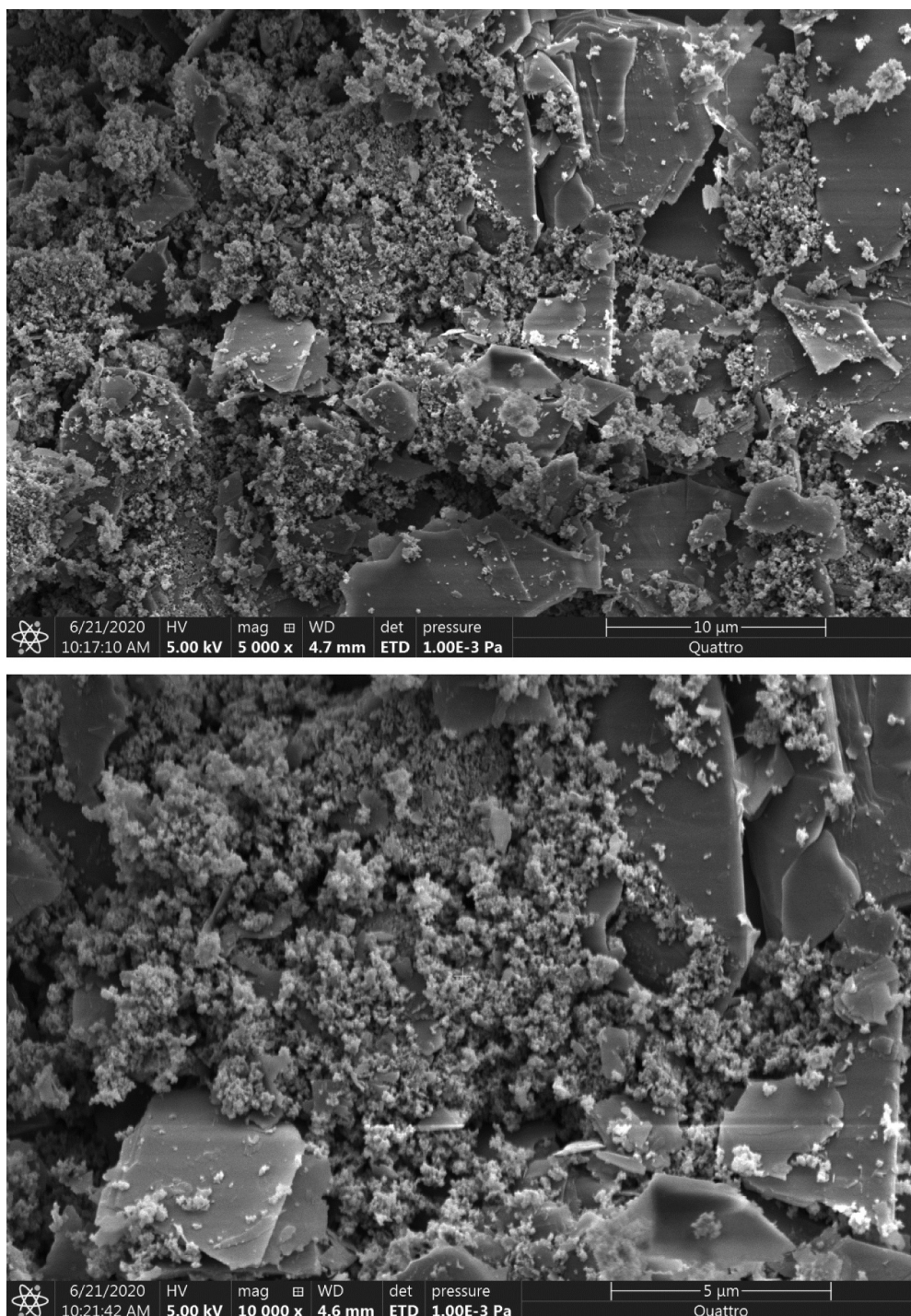


Figure 4. SEM of α -Fe₂O₃/HMS composite.

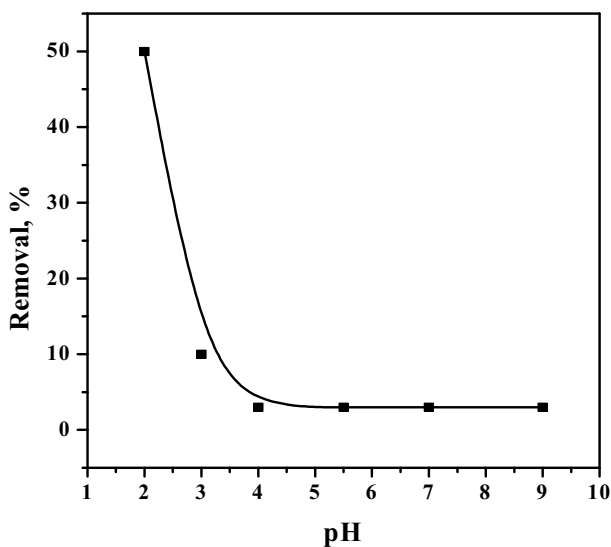


Figure 5. Effect of pH on the sorption of Cr(VI) onto α -Fe₂O₃/HMS composite: [V/m ratio 0.17, T: 25 ± 1°C, [Cr(VI)]: 50 mg/L].

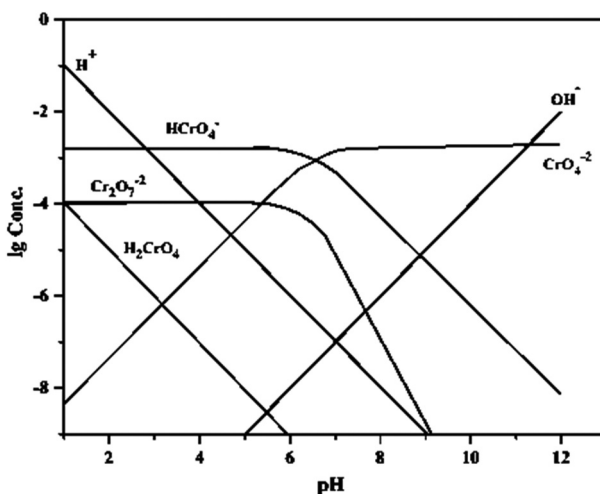


Figure 6. Speciation of Cr(VI) at room temperature and 0.01 ionic strength.

the higher of the adsorption where the equilibrium is attained after 60.0 min. Four kinetic models which defined the adsorption behaviour. The pseudo-first-order kinetics model has been investigated based on Equation (4):

$$\log(q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303}\right)t \quad (4)$$

The pseudo-second-order mechanism according to:

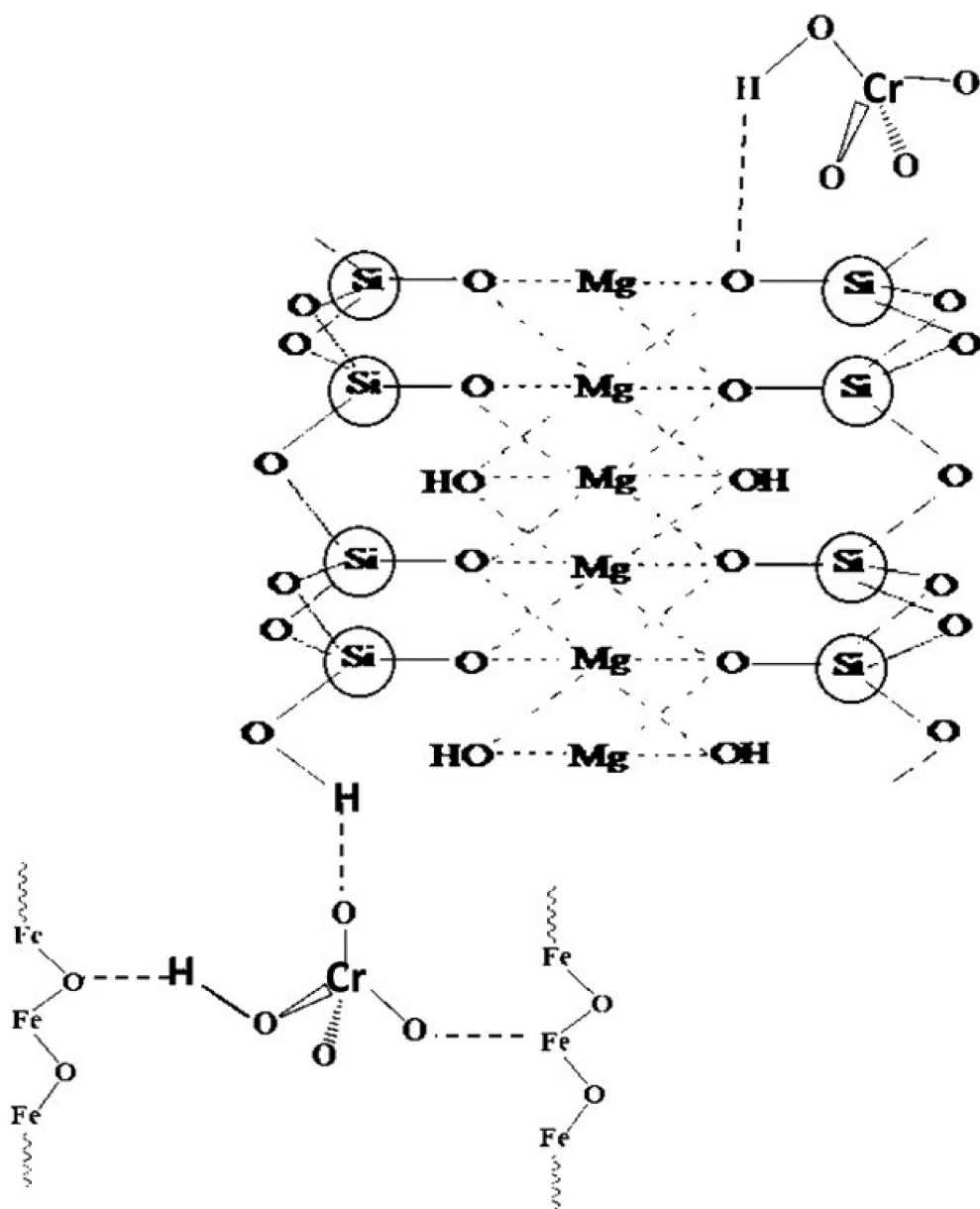


Figure 7. The interactions between Cr(VI) and α -Fe₂O₃/HMS composite.

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

where q_e and q_t (mg/g) refer the amount of Cr(VI) adsorbed onto α -Fe₂O₃/HMS composite at equilibrium and time (t), respectively, k_1 and k_2 are the rate constants of pseudo-first-order (min^{-1}) and pseudo-second-order, respectively, that were estimated from [Figure 10](#) and summarised in [Table 1](#). The estimated sorption capacity (q_e) for equilibrium is close to the experimental results, which means that the data obtained match this model.

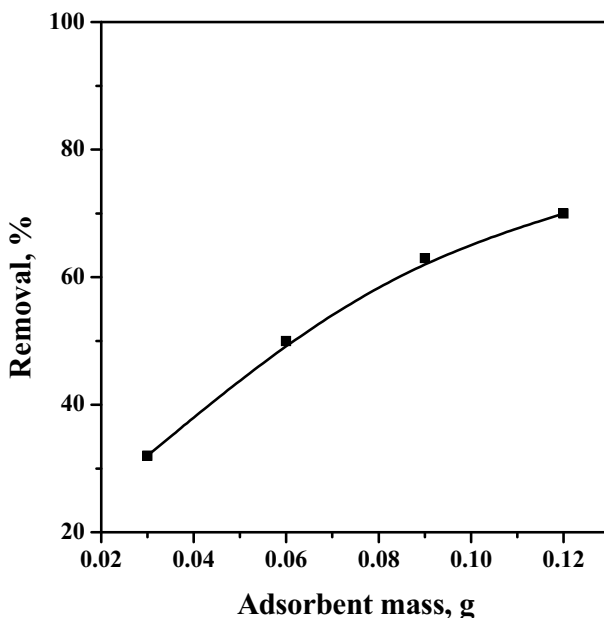


Figure 8. Effect of adsorbent dose on the sorption of Cr(VI). [pH: 2, T: $25 \pm 1^\circ\text{C}$, [Cr(VI)]: 50 mg/L].

The intraparticle diffusion model assumes that the removal process is diffusion-controlled and expressed by the equation:

$$q_t = k_i t^{0.5} + C \quad (6)$$

Where k_i ($\text{mg g}^{-1} \text{min}^{-0.5}$) is the intra-particle diffusion rate constant and C is proportional to the boundary layer thickness. The plots of q_t (mg/g) against $t^{0.5}$, Figure 10 provides a linear relationship that does not cross the point of origin. This implies that the intraparticle diffusion in the adsorption process is not the main mechanism, the parameters are described in Table 1.

Elovich kinetic model emphasises the activated chemisorptions. In Elovich equation

$$q_t = 1/b \ln(ab) + 1/b \ln t \quad (7)$$

The constant (a) is known as the original adsorption rate (mg/g min) and (b) is a constant, the q_t against $\ln t$ plot will evaluate the constants of the model, Figure 10 and the parameters of a and b are described in Table 1.

3.5. Impact of Cr(VI) initial concentration

The impact of Cr(VI) initial concentration was examined in the range 10–50 mg/L using $\alpha\text{-Fe}_2\text{O}_3/\text{HMS}$ composite at 298°K , Figure 11. As the initial concentration of Cr(VI) ions increases, the amount adsorbed (q) slightly increased. This can be due to the existence of ample active centres that are easy for the adsorbate to occupy at low adsorbate concentration. At greater concentrations, however, the active adsorption centres become filled by the binding centres in addition to the coulombic interaction between adsorbed ion negative charges [64].

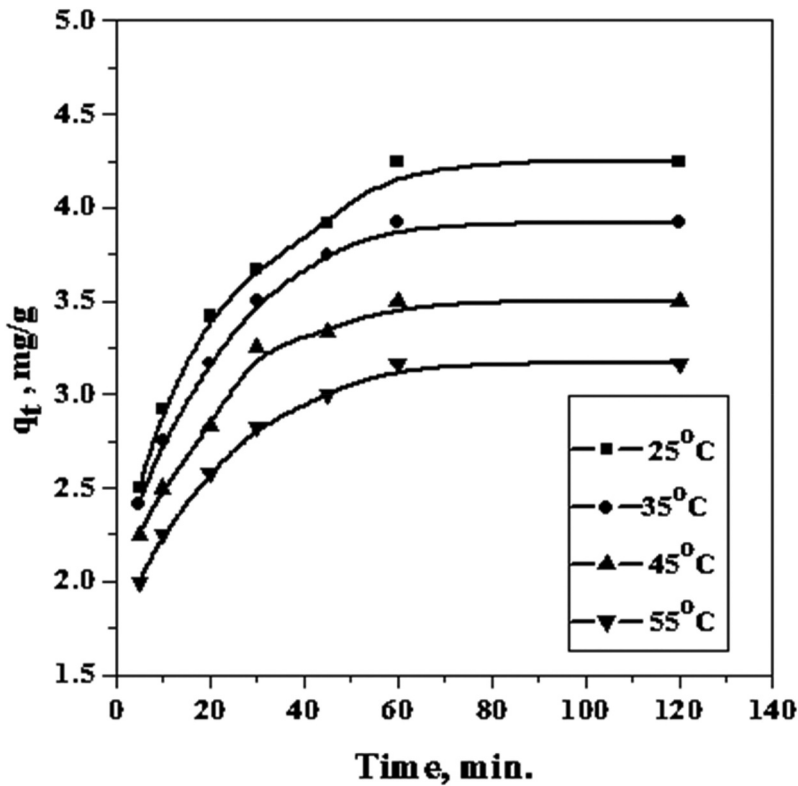


Figure 9. Effect of contact time on percentage of Cr(VI) removal onto α -Fe₂O₃/HMS composite: [V/m ratio 0.17, pH: 2, T: 25 ± 1°C, [Cr(VI)]: 50 mg/L].

3.5.1. Isotherm models

In this investigation, Langmuir and Freundlich models were tested to find the best fitting. In Langmuir isotherm model, the linear form is represented according to the following equation:

$$\frac{C_e}{q_e} = \frac{1}{Q_{\max}b} - \frac{1}{Q_{\max}}C_e \quad (8)$$

where Q_{\max} and b are Langmuir constants assigned to the adsorption quantity and binding energy between the adsorbent and the adsorbate, respectively and can be evaluated from plotting of C_e/q_e against C_e . The results are illustrated in [Figure 12](#) and [Table 2](#). The regression coefficients were around 0.776. Separation factor or equilibrium parameter R_L is derived from Langmuir isotherm and can be calculated from the following equation:

$$R_L = \frac{1}{1 + bc_0} \quad (9)$$

The value of R_L was found to be 0.465; i.e. $0 < R_L < 1$ that means the sorption process is favourable, [Table 2](#). The Freundlich model proposed that the removal process occurred on a heterogeneous-surface. The linear equation is written as:

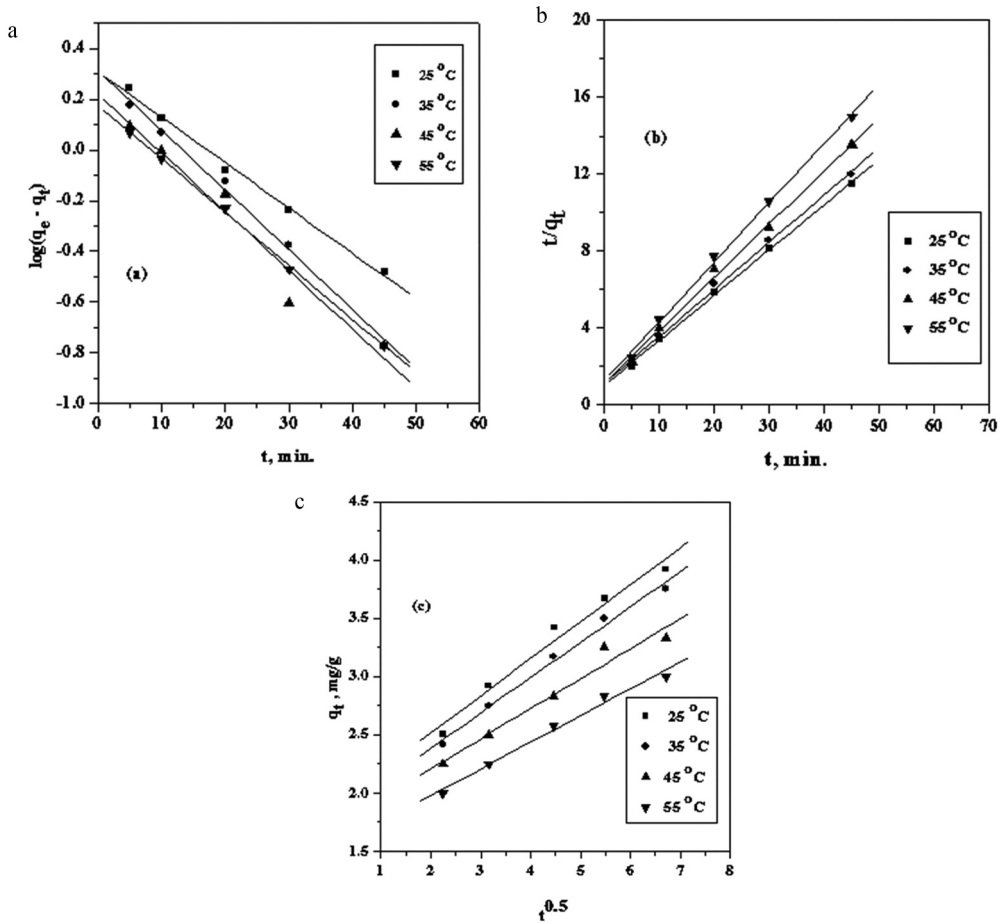


Figure 10. Kinetic models for adsorption of Cr(VI) onto α -Fe₂O₃/HMS (a)Pseudo-first-order model, (b) Pseudo-second-order model, (c) Intraparticle diffusion kinetic model, (d) Elovich kinetic model.

Table 1. The calculated parameters of the pseudo first-order, pseudo second-order, intra particle diffusion and Elovich kinetic models of Cr(VI) onto HMS/ α - Fe₂O₃ composite .

| Model | parameters | 298 K | K308 | 318 K | 328 K |
|---------------------------|--|-------|-------|-------|-------|
| First-order kinetic | $k_1 (\text{min.}^{-1})$ | 0.041 | 0.054 | 0.053 | 0.046 |
| | $q_e, \text{calc.} (\text{gmg}^{-1})R^2$ | 2.027 | 2.056 | 1.656 | 1.503 |
| Second-order kinetic | $K_2 (\text{min.}^{-1})$ | 0.994 | 0.996 | 0.958 | 0.998 |
| | $q_e, \text{calc.} (\text{gmg}^{-1})R^2$ | 4.230 | 4.246 | 3.623 | 3.23 |
| | $q_e, \text{exp.} (\text{mg g}^{-1})$ | 0.996 | 0.996 | 0.996 | 0.998 |
| Intra- particle diffusion | $k_{id}(\text{mg g}^{-1} \text{min}^{-0.5})$ | 4.25 | 3.92 | 3.5 | 3.17 |
| | C | 0.318 | 0.302 | 0.303 | 0.257 |
| | R^2 | 1.883 | 1.781 | 1.781 | 1.697 |
| Elovich | $a, (\text{mgmin}^{-1})$ | 0.987 | 0.995 | 0.995 | 0.983 |
| | $b(\text{gmg}^{-1})$ | 5.845 | 5.731 | 6.917 | 6.288 |
| | R^2 | 1.524 | 1.621 | 1.908 | 2.141 |
| | | 0.998 | 0.992 | 0.966 | 0.992 |

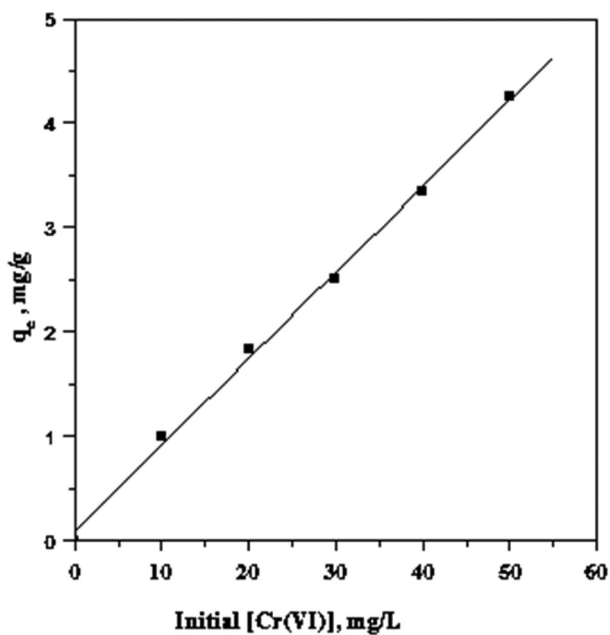


Figure 11. Effect of initial concentration of Cr(VI) on its sorption onto α -Fe₂O₃/HMS: [V/m ratio 0.17, T: 25 °C±1°C, pH: 2.0].

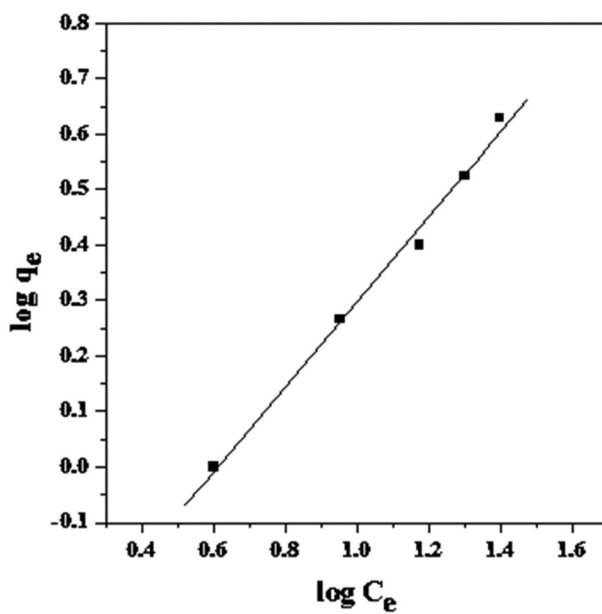
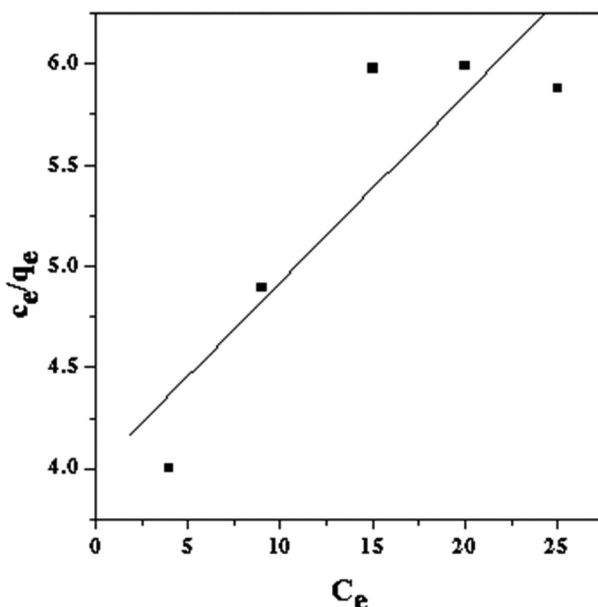


Figure 12. Langmuir Plots for the adsorption of Cr(VI) onto α -Fe₂O₃/HMS: [V/m ratio 0.17, T: 25 °C±1°C, pH: 2.0].

Table 2. Langmuir and Freundlich constants for Cr(VI) adsorption onto HMS/ α -Fe₂O₃ composite.

| Model | Parameters | Value |
|------------|--|-------|
| Langmuir | Qmax(gmg ⁻¹) | 10.75 |
| | b(L/g) | .0230 |
| | R _L | 0.465 |
| | R ² | 0.776 |
| Freundlich | 1/n | 0.766 |
| | K _f (gmg ⁻¹)(L/mg) ^{1/n} | 0.340 |
| | R ² | 0.992 |

**Figure 13.** Freundlich Plots for the adsorption of Cr(VI) onto α -Fe₂O₃/HMS: [V/m ratio 0.17, T: 25 °C±1°C, pH: 2.0].

$$\log q_e = \log K_f + (1/n) \log C_e \quad (10)$$

where K_f and n are the adsorption performance and intensity, respectively, and can be estimated from plotting $\log q_e$ against $\log C_e$, Figure 13; and their values are given in Table 2. The regression coefficients were around 0.992 and the value of $1/n < 1$ (0.14) that shows favourable multilayer adsorption of Cr(VI) and Freundlich isotherm is more adequate than the Langmuir isotherm.

3.6. The effect of temperature

The temperature is a primary parameter that influences the removal process. Raising the temperature leads to increasing the diffusion rate of the adsorbate species through the external boundary layer. In addition, changing the temperature will change the equilibrium capacity of the sorbent. The influence of temperature was tested in the range of

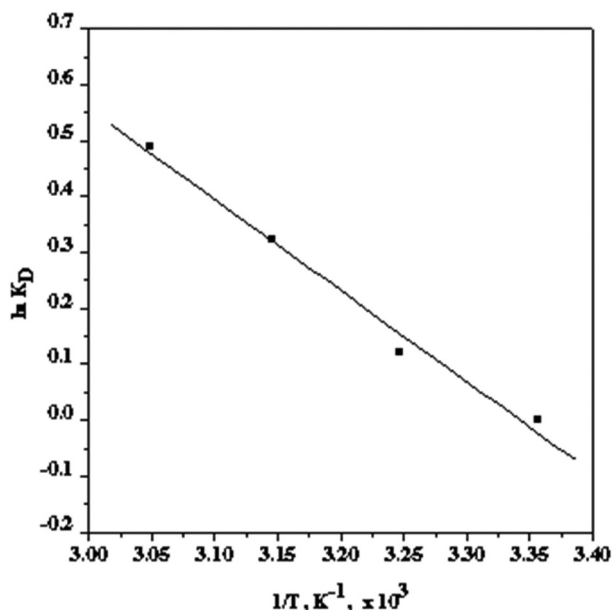


Figure 14. A Plot of $\ln K_D$ against $1/T$ for the adsorption of Cr(VI) onto $\alpha\text{-Fe}_2\text{O}_3/\text{HMS}$: [V/m ratio 0.17, T: $25 \text{ }^\circ\text{C} \pm 1^\circ\text{C}$, pH:2.0].

Table 3. Thermodynamic parameters for sorption of Cr(VI) onto HMS/ $\alpha\text{-Fe}_2\text{O}_3$ composite.

| T, °K | ΔH° (KJ/mol) | ΔS° (J/mol) | ΔG° (KJ/mol) |
|-------|---------------------------|--------------------------|---------------------------|
| 298 | -13.52 | 45.18 | -26.98 |
| 308 | | | -27.44 |
| 318 | | | -27.89 |
| 328 | | | -28.34 |

25–55 °C by drawing K_D versus $1/T$, Figure 14. The thermodynamic parameters can be estimated from the following equations:

$$\ln K_D = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (11)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (12)$$

The values of these parameters are listed in Table 3. The negative ΔH° value suggests the exothermic character of the process and the negative value of ΔG° indicates that the process is spontaneous; the positive entropy change (ΔS°) indicates that the sorption accompanies with increasing the randomness of the system.

A comparison of the adsorption performance of $\alpha\text{-Fe}_2\text{O}_3/\text{HMS}$ composite with other adsorbents is reported in Table 4. The results implied that $\alpha\text{-Fe}_2\text{O}_3/\text{HMS}$ composite can be used efficiently for the uptake of Cr(VI) from aqueous medium.

Table 4. Comparison of sorption capacities for Cr(VI) using various adsorbent materials.

| Sorbent | Q_{max} , mg g ⁻¹ | Optimum pH | Ref. |
|---|--------------------------------|------------|--------------|
| CTAB modified graphene | 21.57 | 2.0 | [65] |
| Modified oak sawdust | 1.70 | 4.0 | [66] |
| MWCNTs | 4.26 | 2.05 | [67] |
| Coconut tree sawdust | 3.46 | 3.0 | [68] |
| Bael fruit | 17.27 | 2.0 | [69] |
| Modified wheat bran | 5.28 | 2.2 | [70] |
| Spent activated clay | 1.42 | 2.0 | [71] |
| Fe ₃ O ₄ @n-SiO ₂ NPs | 3.78 | 2.0 | [72] |
| Fe ₃ O ₄ -coated perlite | 8.77 | 2.0 | [73] |
| α -Fe ₂ O ₃ /HMS composite | 10.75 | 2.0 | [This study] |

4. Conclusion

Effectively, α -Fe₂O₃/HMS composite can be utilised to removal Cr(VI) from aquatic environment. Pseudo-second-order model refers to uptake of Cr(VI) ions. The Freundlich model gives the experimental data a considerably better match. The adsorption process was found to be exothermic, spontaneous with an increase in system randomness.

Acknowledgments

The authors would like to extend their appreciation to the Chemistry department, Jouf University for giving the permission to access the analytical equipment.

Disclosure statement

No potential conflict of interest was reported by the authors

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