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DESIGN AND ADSORPTION PROPERTIES OF IRON OXIDE/COMMERCIAL POLYMER COMPOSITE

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Waste water treatment is particularly i mportant from an environmental perspective. In this study, industrially produced Duolite ion-exchange resin was modified with iron oxides, and the resulting composite was used to adsorb chromium(VI) ions from aqueous solutions.

The composite was produced using co-precipitation of iron salts in the presence of the ion-exchange resin. The effect of the initial concentration of the metal ion in the solution on the process was determined.

Based on theexperimental results, sorption isotherms were plotted, and Langmuir, Freundlich, Dubinin-Radushkevich, Temkin and Halsey models were applied. It was found that the Freundlich and Halsey models describes the adsorption better. The correlation coefficient for both models is 0.9866. The Freundlich isotherm constant is 2.38 (mg/g)·(l/mg)1/n; the non-uniformity coefficient is 0.8452. This result indicates that the process occurs at energetically different active centers, which is related to the structureof the composite.

Keywords: ion-exchange resin, modification, adsorption, isotherm models

INTRODUCTION

In the modern era, environmental pollution resulting from human activity is considered one of the major environmental problems. Various industrial sectors occupy a prominent place as sources of pollution. For example, dye synthesis, iron and steel production, leather tanning, metal surface cleaning, etc. As a result of these processes, large quantities of heavy metal ions (lead, cadmium, mercury, chromium, nickel, zinc, iron, and others) enter the environment, including water bodies. This leads to serious consequences: the destruction of aquatic ecosystems (eutrophication, oxygen depletion) and chronic diseases in humans (liver and kidney pathologies, cancer, infections) [1].

Trivalent and hexavalent chromium ions are among the heavy metal ions emitted into the environment as a result of various industrial processes. Their content in wastewater can vary from 10 to 1000 mg/L. Hexavalent chromium is toxic, since it is a stronger oxidizer than trivalent chromium, and is classified as a group A carcinogen. In addition, hexavalent chromium compounds easily penetrate into the cells of living organisms. In humans, they are one of the causes of lung and stomach cancer. They can destroy DNA, cause dermatitis,

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kidney disease, damage the liver and nerve tissue, and cause many other diseases [2]. In this regard, the European Union RoHS Directive has introduced restrictions on the use of hexavalent chromium compounds in industry, and in some countries the maximum permissible concentration of chromium(VI) ions in wastewater discharged into the environment is limited to 0.02 mg/L. Therefore, various methods (adsorption, chemical precipitation, electrodialysis, coagulation, reverse osmosis, etc.) are currently used to remove chromium ions from wastewater. Adsorption is considered more promising due to the simplicity of the associated equipment and the cost of the process. On the other hand, wastewater treatment typically involves the use of several methods in sequence, with the final stage often being carried out by adsorption [3]. In recent years, various nanocomposites, activated carbons and other materials for the adsorption of chromium (VI) ions have been considered [4-7].

In this study, a magnetic composite based on a commercial polymer *Duolite* was prepared using a coprecipitation method and investigated as an adsorbent for a toxic metal chromium (VI).

Specifically, various adsorption models were used toinvestigate the adsorption of chromium (VI) ions on the synthesized polymer/iron oxide composite. Composite was abbreviation as IODC.

EXPERİMENTAL

Materials

Reagent used for the composite are the following: Duolite - a commercial polymer with sulfonic acidic groups, chemically pure $FeSO_4 \cdot 7H_2O$, $FeCl_3 \cdot 6H_2O$, NH_4OH. As the source of Cr(VI) ions chemically pure $K_2Cr_2O_7$ was used.

Synthesis of composite

For synthesize the composite, the commercial polymer was placed in a reaction flask, 0.1 M solutions of FeSO4·7H2O and FeCl3·6H2O were added, and the mixture was stirred. The total volume of the solution was 200 ml. Stirring was carriedout using a magnetic stirrer at room temperature for 1 hour. The solution temperature was then raised to 40°C, at which point ammonium hydroxide solution was added dropwise to the reaction flask. The pH of the reaction medium should be 11. Thesuspension was then stirred under a nitrogen atmosphere at 80°C for 1 hour and at room temperature for 3 hours. Finally, the composite was separated, washed sequentially with distilled water, ethanol and acetone, and finally air-dried.

Calibration Curve for Determination of chromium (VI) ions

A UV-Vis spectrophotometer was used to determine the amount of Cr(VI) ions. For this purpose, 0.294 g of potassium dichromate was dissolved in 200 ml of distilled water (stock solution). Various volumes of potassium dichromate were thensampled from the stock solution, added to a 25 ml volumetric flask, and diluted to the mark with distilled water. This resulted in working solutions with concentrations of 0.02, 0.04, 0.06, 0.08, 0.1, 0.12, 0.14, 0.16, 0.18, and 0.2 mM. The optical density of the working solutions was measured using a UV-visible spectrophotometer, and the calibration curve was found to obey the optical density equation $y=10.735x$ ($R^2=0.9983$).

Adsorption experiments

The experiments were carried out in a 100 ml flask. For each experiment, the sample was placed in the flask and 25 ml of Cr(VI) ions solution (at the required concentration) was added. T he suspension was stirred at room temperature for 4 hours. This period was previously established to be sufficient for equilibrium to form in the solid-liquid system. The composite

was then separated from the liquid medium using an external magnet, and the amount of chromium ions in the medium afteradsorption was determined. Based on the results obtained after adsorption, the sorption capacity (q_e)*qe* and sorption efficiency (R)*R* were calculated using the following formulas:

$$q_e = \frac{C_0 - C_e}{g} \cdot V \quad (1)$$

$$R = \frac{C_0 - C_e}{C_0} \cdot 100 \quad (2)$$

Where C_0 is the initial concentration of chromium ions in the solution, mg/l; C_e is the equilibrium concentration of chromium ions in the solution, mg/l; g is the mass of the composite, g; V is the volume of the solution, l.

To determine the adsorption capacity of the sample, the dependence of the sorption capacity on the equilibrium concentration of the metal ion in the solution was established based on the results calculated using formula 1. The experimental resultswere analyzed using various isotherm models.

RESULTS AND DISCUSSION

The presence of iron oxide in the resulting composite was confirmed by the sample's adequate response to an external magnet. The resulting composite is insoluble in water, acidic and alkaline solutions, and organic solvents.

Adsorption studies

It is known that as a result of the adsorption process, adsorbates accumulate either on the surface of the adsorbent or on the interfacial surface. In general, an adsorbate can be attached to an adsorbent due to electrostatic attraction, ion exchange, interaction of ion pairs, van der Waals and cation-π-forces, as well as hydrophobic hydration [8,9]. Adsorption can be described by physicochemical processes. In physical adsorption, adsorption of adsorbents on the adsorbent surface occursdue to weak forces (reversible), whereas in chemical adsorption it occurs due to chemical bonds (mainly irreversible) [10]. According to the literature, adsorption of chromium ions can occur due to electrostatic attraction [11,12], adsorption accompaniedby reduction [13,14], and simple adsorption [15]. The type and nature of the adsorbent and its functional groups determine the mechanism of chromium ion removal.

To study the sorption by the IODC isotherm of the process was analyzed. For this purpose, the dependence of the composite's sorption capacity on the initial concentration of metal ions in the solution was assessed. The initial metal ion concentration varied in the range of 20–200 mg/L. The results are presented in the Table 1.

Table1. Dependence of the sorption capacity and sorption efficiency of the IDOC on the initial concentration of chromium (VI) ions

C_0, mg/l	20	40	60	80	100	120	140	160	180	200
q_e, mg/g	18	32	48	65	80	97	110	120	127	130
R, %	45	40	40	40,63	40	40,42	39,29	37,50	35,28	32,5

As can be seen, with an increase in the initial concentration of chromium (VI) ions in the solution, the sorption capacity of the composite increases to 130 mg/g. It should also be noted that the sorption capacity of the composite does not change at initial concentrations of chromium (VI) ions in the solution above 200 mg/L. This result can be explained by the fact that the composite used has a limited number of active sites, which are filled after a certain initial concentration is reached.

Besides these, with an increase in the initial concentration of chromium (VI) ions from 20 mg/L to 200 mg/L, the sorption efficiency of the composite decreases from 45% to 32.5%. The decrease in sorption efficiency with an increase in the initialconcentration of Cr(VI) ions

is explained by the fact that the composite has a limited number of active sites, and at a certain concentration of metal ions, the active sites become saturated.

A sorption isotherm is a plot of the amount of chromium (VI) ions sorbed by a composite at a constant temperature versus the equilibrium concentration of metal ions in the solution. A sorption isotherm provides information about the process type(chemical or physical sorption), the sorption energy, and the maximum sorption capacity of the adsorbent. The sorption isotherm of chromium (VI) ions by a composite is shown in Figure 1.

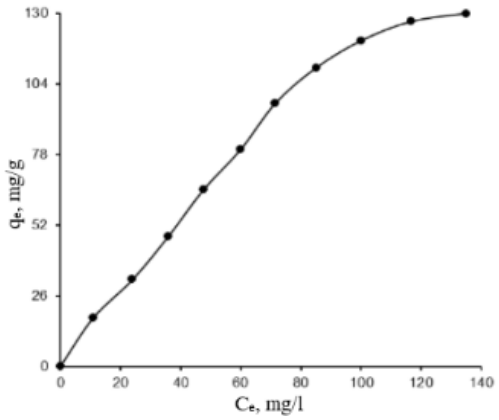

Figure 1. Sorption isotherm of chromium (VI) ions on the composite

Mathematical modeling of sorption isotherms

In this paper, two-parameter isothermal models were used to describe the sorption isotherm: Langmuir, Freundlich, Dubinin-Radushkevich (D-R), Temkin, and Halsey [16,17]. Linear forms of the models (equations (3)–(7), respectively) were usedto calculate the corresponding parameters.

Langmuir model

$$\frac{C_e}{q_e} = \frac{1}{K_L \cdot q_{\max}} + \frac{1}{q_{\max}} \cdot C_e \qquad (3)$$

q_e-the sorption capacity of the composite for Cr (VI) ion at the equilibrium condition, mg/g,
C_e-the concentration of Cr (VI) ion at the equilibrium condition, mg/l,
q_{max}-the maximum monolayer sorption capacity of the composite for Cr (VI) ion, mg/g,
K_L-Langmuir constant, l/mg.

The separation efficiency (R_L) can be calculated using the value of K_L by the following formula: R_L=11+$K_L C_0$*RL=11+KLC0*

Freundlich model

$$logq_e = logK_F + \frac{1}{n} logC_e \qquad (4)$$

K_F-the Freundlich constant, which characterizes the sorption capacity at a given temperature, n- the sorption intensity.

D-R model

$$lnq_e = lnq_{D-R} - k_{D-R} \bullet \varepsilon^2 \qquad (5)$$

q_{D-R} - the theoretical maximum sorption capacity of composite, mg/g,
k_{D-R} - constant related to sorption energy, mol^2/C^2,
ε-Polanyi sorption potential.

Polanyi sorption potential was calculated by the following formula:

$$\varepsilon = RT\ln\left(1+\frac{1}{C_e}\right)$$

where R is the universal gas constant (R=8.314 C/mol·K), T is the absolute temperature (K).

The average free energy of the sorption process (kC/mol) was calculated by using value of the KD-R parameter the subsequent equation $E=1/\sqrt{2kD-R}$

For calculation the parameters of the models, the following graphs were constructed.

Temkin model

$$q_e = \frac{RT}{b_T} lnA_T + \frac{RT}{b_T} lnC_e \quad (6)$$

where R is the universal gas constant (R=8.314 C/mol·K), T is the absolute temperature (K).
A_T - Temkin isotherm constant theoretical maximum sorption capacity of composite, mg/g,
B_T - *Temkin isotherm constant related to the sorption heat*,
b_T – Temkin constant related to adsorption potential.
The parameter B_T is calculated as follows:

$$B_T = \frac{RT}{b_T}$$

Halsey model

$$lnq_e = \left(\frac{1}{n_H}\right) lnK_H - \left(\frac{1}{n_H}\right) ln\left(\frac{1}{C_e}\right) \quad (7)$$

where n_H and K_H are the Halsey isotherm constants.

Then, based on the experimental results obtained during the sorption of chromium(VI) ions by the composite, corresponding graphs were plotted for the aforementioned isotherm models (Figures 2).

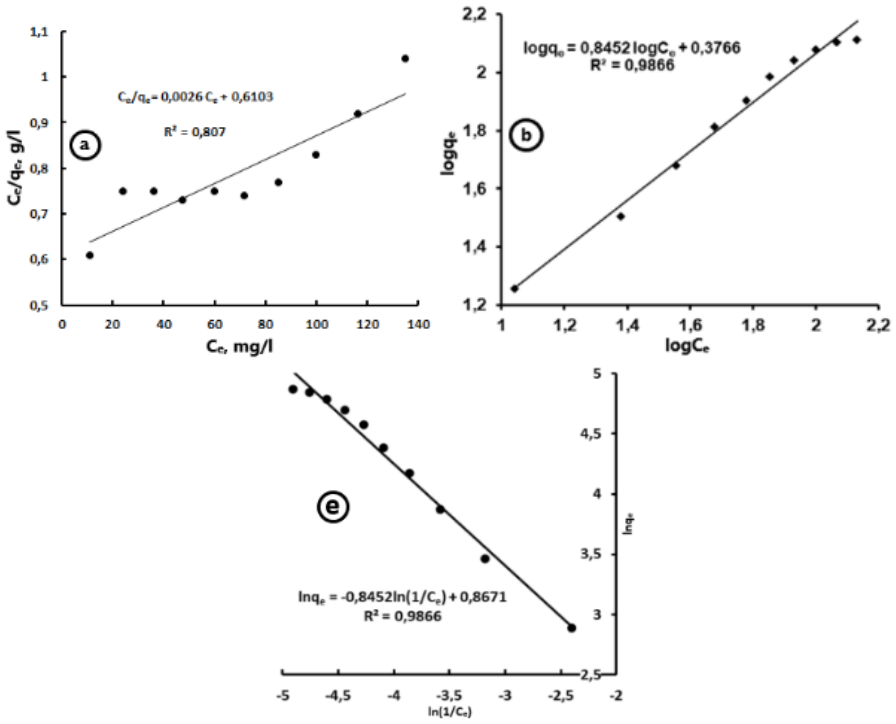

Figure 2. Adsorption isotherm plots inlinear forms: (a) Langmuir isotherm model, (b) Freundlich isotherm model, (c) Dubinin-Radushkevich isotherm model, and (d) Temkin isotherm model and (e) Halseymodel

Then, the corresponding parameters were calculated, that was presented in table 2.

Table 2. Parameters of adsorption of chromium ions from aqueous solutions by IODC

Parameter	**Langmuir**	**Freundlich**	**D-R**	**Temkin**	**Halsey**
q_{max}, mg/g	384.62	-	-	-	-
K_L, l/mg	0.00426	-	-	-	-
R_L	0.54÷0.93	-	-	-	-
K_F, $(mg/g)\cdot(l/mg)^{1/n}$	-	2.38	-	-	-
1/n	-	0.8452	-	-	-
q_{D-R}, mg/g	-	-	92.57	-	-
k_{D-R}, mol^2/C^2	-	-	$4\cdot10^{-5}$	-	-
E, kC/mol	-	-	0.112	-	-
B_T	-	-	-	50.603	-
b_T, C/mol	-	-	-	48.96	-
A_T, l/g	-	-	-	0.093	-
n_H	-	-	-	-	1.18
K_H	-	-	-	-	2.80
R^2	0.807	0.9866	0.7139	0.9453	0.9866

As can be seen from the table, the R_L parameter of the Langmuir model ranges from 0 to 1. This indicates that the resulting composite is considered favorable for chromium ions. On the other hand, the correlation coefficient is 0.807 (relatively low), indicating that this model does not fully describe the sorption process. The fact that the value of 1/n, the parameter of the Freundlich model, is less than 1 also indicates that the composite is favorable for sorption. The activation energy calculated usingthe Dubinin-Radushkevich model is very low (0.112). This indicates that chromium ions are adsorbed by the composite in pores of varying sizes or on the surface. The low correlation coefficient (0.7139) also confirms this. In general, the highestcorrelation coefficient was obtained for the Freundlich and Halsey models. This indicates that chromium ion sorption occurs predominantly in heterogeneous active sites.

CONCLUSION

In this study, The iron oxide synthesis reaction was carried out using iron(II) sulfate, iron(III) chloride, and sodium hydroxide via a hydrothermal method in the presence of a Purolite ion-exchange resin. As a result, a Purolite/iron oxide composite was developed and produced. To evaluate the composite's sorption capacity for chromium(VI) ions, the dependence of the sorption capacity and degree of sorption on the initial concentration was studied. It was found that, with an initial chromium(VI) ion concentration varying in the range of 20-200 mg/L, the sorption capacity of the composite was 18-130 mg/g, and the degree of sorption was in the range of 32.5-45.0%. The sorption isotherm constructed based on these results was processed using two-parameter isothermal models. It was established that sorption occurs in energetically heterogeneous active centers.

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