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Abstract: A Fe-modified pozzolane was prepared and employed for the removal of CrO_4^{2-} ions from aqueous solution under batch type experiments as a function of contact time, initial concentration of metal ion and temperature. The adsorption isotherms are described by means of Freundlich, Langmuir, and Dubinin-Radushkevich (D-R) models. The pozzolane was characterized by XRD diffraction analysis. The results showed that the maximum adsorption capacity of Fe-modified pozzolane for CrO_4^{2-} ions was $(3.23 \pm 0.01) \times 10^{-3}$ mmol g^{-1}. The adsorption was found to be initial concentration and temperature dependent. The thermodynamic parameters values such as ΔH^0, ΔG^0, and ΔS^0 were obtained to predict the nature of adsorption. These values show that the adsorption reaction is endothermic and spontaneous. The results show that the Fe-modified pozzolane, that is an easily available material, can be successfully used as adsorbent of anionic species, such as CrO_4^{2-} ions in aqueous solutions, and can be an alternative for more costly adsorbents used for chromates removal in wastewater treatment processes.

Keywords: Adsorption, chromate ions, Fe-modified pozzolane, isotherms, thermodynamics parameters

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INTRODUCTION

Different investigation works have been reported for the elimination of toxic metals, such as arsenic, copper, lead, zinc, and others (1–3). Many industries produce big volumes of wastewater which contain polluting metals such as chromium, a very toxic metal and difficult to degrade when found as Cr(VI). Chromium is an active metal that exists in several oxidation states, the most stable in aqueous solution being the hexavalent, Cr(VI), and the trivalent, Cr(III); however, the distribution of Cr(VI) species, as CrO_4^{2-} ions, in the environment causes a very harmful contamination (4). Therefore the separation of chromium from industrial wastes to avoid the contamination of water bodies and soil fractions caused by CrO_4^{2-} ions is a major concern. For the treatment of industrial waste, several methodologies such as adsorption have been used. Many studies have shown that the metallic oxides used as sorbentes are very useful for the extraction and recovery of many metallic ions thanks to their great thermal stability, high chemical resistance, and low solubility in a wide pH interval. Thus, pozzolane, a solid rich in silicates and aluminates, may be useful for the removal of metals in water. As advantages, this material is abundant in nature and has a very low cost. The pozzolane has the capacity of charging its surface electrically, a fact that confers to it a capacity of ionic exchange. When iron ions are adsorbed on its surface, Fe-modified pozzolane gets the capacity to retain anions such as chromates present in aqueous effluents; for example, those coming from the tanner companies (5–8). Many investigations have been carried out to examine the adsorption and transport properties of many radionuclides and other adsorptive substances on different adsorbents (9–17). In those studies, adsorptions of cations and anions on inorganic materials were examined as a function of time, cation concentration, and temperature. However, the kinetics and thermodynamics parameter of anion adsorption have not been thoroughly studied, specifically the kinetic and thermodynamic parameters of chromate anion adsorption on Fe-modified pozzolane, a novel modified material, have not been reported in the specialized literature. Thus, the aim of this work is to study the CrO_4^{-2} ions adsorption behavior from aqueous solution on Fe-modified pozzolane, as a function of shaking time, initial concentration, and temperature to test isotherm models and to investigate the thermodynamic parameters to predict the nature of adsorption.

EXPERIMENTAL

Materials

The following analytic grade materials were used without further purification: iron chloride hexahydrate $FeCl_2.6H_2O$ A.C.S. reagent (J. T.

Baker, 100.1% wt purity), potassium chromate K_2CrO_4 A.C.S. reagent (Merck, 99.5% wt purity), and silver nitrate $AgNO_3$ A.C.S. reagent (Merck, 99.5% wt purity).

Adsorbent Preparation

To prepare Fe-modified pozzolane, 10 g of natural pozzolane from Los Romanes, Aguascalientes, México were put in contact with 250 mL of 0.1 eq L^{-1} $FeCl_3$ aqueous solution in a 500 mL rounded glass flask. The suspension was refluxed for 8 h. At the end of this time, the brown-red suspension was let to cool to room temperature and then the suspension, together with a dark-red residue, was centrifuged for 2 h to recover the solid phase. To eliminate the chloride ions, the solid phase was washed with distilled water and the suspension was centrifuged again. After 2 h of centrifuging, the supernatant was discarded. These operations were repeated several times until chloride ions were not detected as AgCl in the discarded liquid. To dry the obtained Fe-modified pozzolane, the centrifuge tubes with the solid were put into a boiling water bath. Later, the recovered Fe-modified material was dried again at 343 K for 24 h, and then was thermally treated at 473 K for 5 h.

Adsorbent Characterization

The Fe-modified pozzolane was characterized by X-ray diffraction using a Siemens D500 diffractometer coupled to a copper anode X-ray tube, the wavelength K_α was selected with a diffracted beam monochromator. The X-ray tube was operated at 35 kV and 20 mA. Diffraction pattern was obtained in scanning mode with a 0.02° (2θ) step size. The compound was identified comparing the diffraction pattern with the JCPDS cards in the conventional way. The BET surface area of the Fe-modified pozzolane was determined by the standard multipoint technique with a Micromeritics Gemini 2360, the sample was pretreated at 473 K for 2 h in N_2 atmosphere.

Adsorption Experiments

Batch type experiments were carried out at 293 K to determine the kinetic removal of CrO_4^{2-} ions by shaking in closed vials 100 mg of Fe-modified pozzolane (adsorbent) and 10 mL aliquots of 1.0×10^{-4} mol L^{-1} CrO_4^{2-} ions solution with a pH 5.5, for different time intervals (5 min to 24 h) to attain equilibrium distribution. Later, the samples were centrifuged to recover the liquid phase. All of the experiments were performed in triplicate, by running three parallel independent closed vials, simultaneously.

The adsorption capacity of CrO_4^{2-} ions by the Fe-modified pozzolane was determinated from the difference between the initial and final concentrations of CrO_4^{2-} ions in the aqueous solutions before and after adsorption, using a Shimadzu ultraviolet-visible 265 spectrophotometer analyzer at $\lambda = 370\,\text{nm}$. The amount of CrO_4^{2-} ions adsorbed per unit mass of the adsorbent and the percent adsorption of CrO_4^{2-} ions were calculated by:

$$q = \frac{(C_o - C_e)V}{m} \tag{1}$$

$$\%Ads = \frac{(C_i - C_f)}{C_i} \times 100 \tag{2}$$

were, q is the amount of CrO_4^{2-} ions adsorbed on Fe-modified pozzolane (mmol g^{-1}), C_o and C_e are the initial and equilibrium concentration of solution (mol/L), C_i and C_f are the initial and final solution phases concentration (mol L^{-1}), V is the volume of solution (L), and m is amount of adsorbent (g). For the chromate concentration studies, solutions with the desired concentrations (10^{-4}–10^{-5} mol L^{-1}) of CrO_4^{2-} ions were prepared by successive dilutions of a stock solution of 1.0×10^{-4} mol L^{-1} K_2CrO_4 in distilled water.

RESULTS AND DISCUSSION

Adsorbent Characterization

Pozzolane X-ray diffraction patterns showed that this material is a complex mixture of quartz (64.36%), disorded albite (19.55%), sanidine (10.73%), lawrencite (2.31%), halite (1.22%), and gypsum (1.83%). The Fe-modified pozzolane, as obtained in our laboratory, has mainly two components, quartz (file JCPDS 33-1161) and gismondine (file JCPDS 33-1161). Albite (file JCPDS 20-0548) and lawrencite (file JCPDS 1-1106) were also found in the Fe-modified samples. The surface area found by BET method for Fe-modified pozzolane, was $457\,m^2\,g^{-1}$, which indicates that the material has a high surface area, ideal for adsorption purposes.

Adsorption Kinetic and Effect of Initial Concentration

The initial pH value of the chromate aqueous solution was 5.5. The adsorption capacities of the iron modified pozzolane were studied at this pH value. Figure 1 shows fast chromate ions adsorption rate at the beginning of the process and that the equilibrium is reached in about

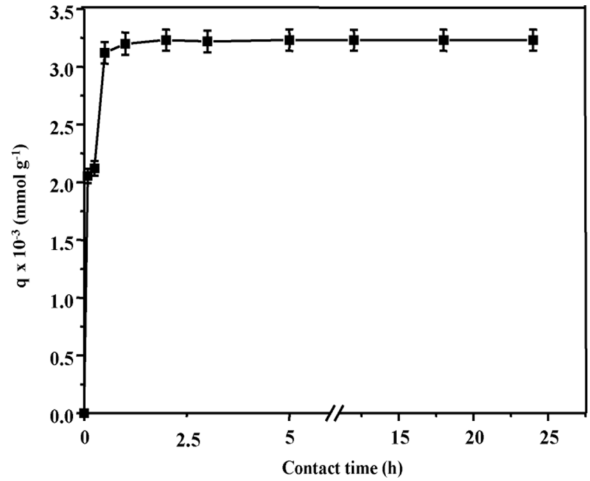

Figure 1. Effect of contact time of CrO_4^{2-} ions adsorption on Fe-modified pozzolane.

2 h. The adsorption at equilibrium for CrO_4^{2-} ions by Fe-modified pozzolane was found to be $(3.23 \pm 0.01) \times 10^{-3}$ mmol g^{-1}. On the other hand, the effect of chromate ion concentration on its adsorption on Fe-modified pozzolane was studied by varying the adsorptive concentrations (10^{-5}–10^{-4} mol L^{-1}), at 293 K and for 2 h of shaking time. The results are shown in Fig. 2. Adsorption of CrO_4^{2-} ions is highly concentration dependent. As it is seen the percentage of adsorption decreased as the initial concentration of CrO_4^{2-} ions increase. Therefore the percent adsorption is higher in the more diluted chromate solutions, which is due to the fact that there are more surface active sites per gram of adsorbent available for deposition of a lesser number of adsorbing species at higher dilution.

Upon comparing the adsorption capacity (3.75 mg g^{-1}) of Fe-modified pozzolane to the corresponding values of other materials such as calcareous karst soil, 0.042 mg g^{-1} (18); surfactant modified clinoptilolite, 12.3 mg g^{-1} (19); activated charcoal, 12.87 mg g^{-1} (20) and activated alumina, 7.44 mg g^{-1}(20), it can be seen that the Fe-modified pozzolane shows an equilibrium adsorption capacity higher than that of the calcareous karst soil value and similar to the values showed by the other mentioned materials.

Adsorption Isotherms

Concentration data were analyzed for Freundlich, Langmuir, and Dubinin-Radushkevich (D-R) adsorption isotherms. The Freundlich

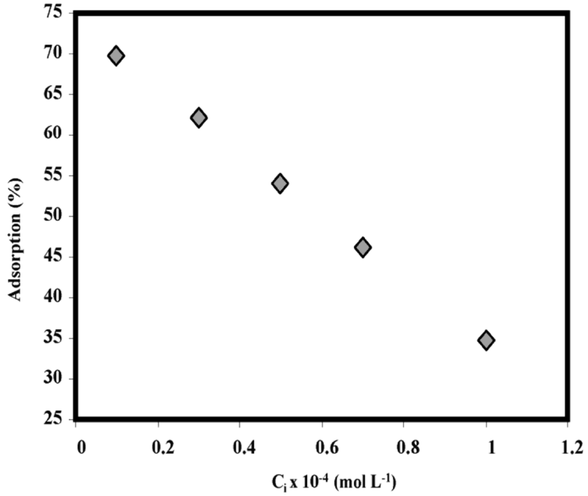

Figure 2. Effect of initial CrO_4^{2-} ions concentration on its adsorption.

model assumes a heterogeneous adsorption surface with sites that have different energies of adsorption and are no equally available. The Freundlich isotherm is more wide used but provides no information on the monolayer adsorption capacity in contrast to the Langmuir model. On the other hand, it was observed that the data fitted well to the classical linearized Freundlich equation [Eq. (3)].

$$\log a_e = \frac{1}{n}\log C_e + \log K_F \tag{3}$$

where a_e and C_e are the amount of CrO_4^{2-} ions adsorbed (mol g^{-1}) and bulk concentration (mol L^{-1}) at equilibrium respectively and $1/n$ and K_F are the Freundlich constants referring to adsorption capacity and adsorption intensity, respectively. The plot of log a_e vs. log C_e is given in Fig. 3, which is a straight line with a correlation coefficient $R^2 = 0.9972$, the result suggest a fair validity of the Freundlich isotherm over the entire range of chromates ion concentrations (10^{-5}–10^{-4} mol L^{-1}) at equilibrium. From the slope and intercept of the plot, the numerical values of the Freundlich constants, i.e., $1/n$ and K_F were determined and the values obtained were 0.67 ± 0.01 and $(3.58 \pm 0.05) \times 10^{-3}$ mol g^{-1} respectively. The value $1/n$, less than unity, is attributed to a heterogeneous surface structure of the adsorbent and to the adsorbed species which have no appreciable interaction among them. The fractional values of $1/n$ also indicate an exponential distribution of energy sites (21). In general, as the K_F values increases, the adsorption capacity of the adsorbent, for the

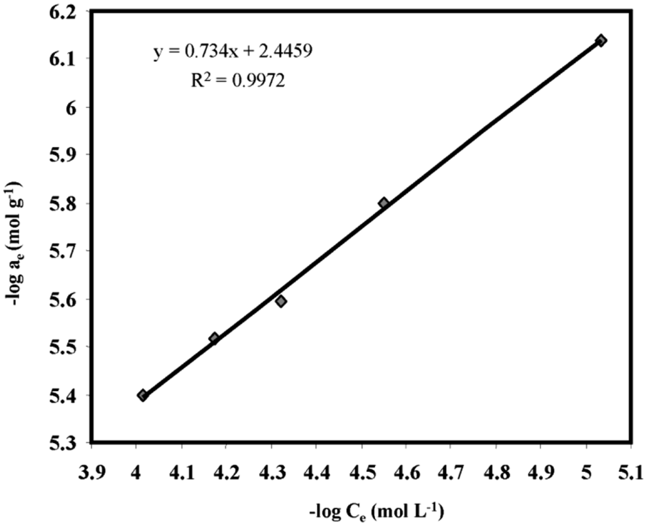

Figure 3. Freundlich adsorption isotherm of CrO_4^{2-} ions adsorption on Fe-modified pozzolane.

given adsorbate, increases. *n* values between 1 and 10 show beneficial adsorption. On the other hand, observed value of K_F suggests a significant affinity of CrO_4^{2-} ions towards the Fe-modified pozzolane.

The adsorption data were also tested using Langmuir isotherm in the following linearized form:

$$\frac{C_e}{a_e} = \frac{1}{ka_{\max}} + \frac{C_e}{a_{\max}} \tag{4}$$

where C_e is the equilibrium concentration of CrO_4^{2-} ions in solution (mol L^{-1}), a_e is the amount of CrO_4^{2-} ions adsorbed (mol g^{-1}) and k and $a_{\max}$ are Langmuir constants related to the adsorption energy and the maximum adsorption capacity respectively. A linear plot was obtained when C_e/a_e was plotted against C_e (Fig. 4) over the entire range of metal ion concentration at equilibrium investigated. The slope of this plot gives the value of $a_{\max} = (5.78 \pm 0.01)\times10^{-6}$mol g^{-1} and intercept yields the value of $k = (1.58 \pm 0.3)\times10^{6}$ L mol^{-1}, with a correlation coefficient $R^2 = 0.9937$.

In order to understand the nature of the adsorption processes, the D-R isotherm model was also verified in the linearized form (22):

$$\ln C_e = \ln a_{\max} - K\varepsilon^2 \tag{5}$$

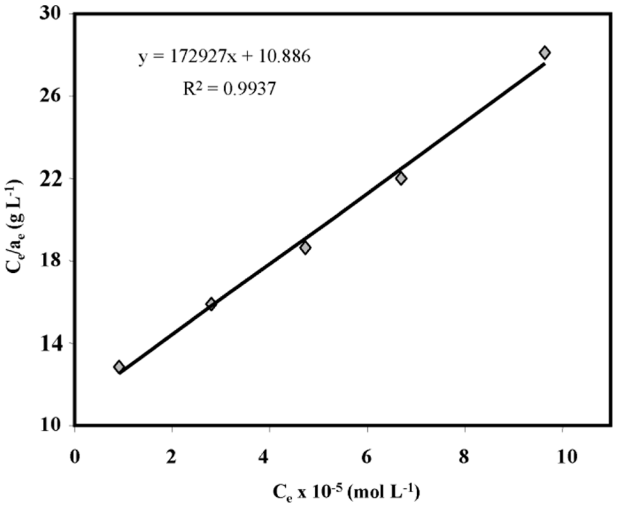

Figure 4. Langmuir isotherm of CrO_4^{2-} ions adsorption on Fe-modified pozzolane.

where C_e is the amount of CrO_4^{2-} ions adsorbed at equilibrium on Fe-modified pozzolane surface and a_{max} is the maximum amount of CrO_4^{2-} ions that can be adsorbed on Fe-modified pozzolane under the optimized experimental conditions. K is a constant related to ion adsorption energy ($mol^2\ kJ^{-2}$) and ε is the Polanyi potential ($kJ\ mol^{-1}$) which is calculated from:

$$\varepsilon = RT\ln(1 + \frac{1}{C_e}) \tag{6}$$

where R is the gas constant, $8.314\ J\ mol^{-1}K^{-1}$, T is the absolute temperature in Kelvin degrees, and C_e is the concentration in solution at equilibrium ($mol\ L^{-1}$). A plot of ln a_e vs. ε^2 is shown in Fig. 5, which is a straight line. It indicates that the D-R isotherm is obeyed by the adsorption data over the entire concentration of CrO_4^{2-} ions at equilibrium studied. The values of maximum adsorption found, $a_{max} = (7.55 \pm 0.05) \times 10^{-5}\ mol\ g^{-1}$ have been assessed from the intercept and slope of the linear plot shown in Fig. 5, and the correlation coefficient for this isotherm was 0.9989. The mean free energy of adsorption E, which is defined as the free energy of transfer of one mole of solute from infinity (in solution) to the surface of iron modified pozzolane, has been evaluated using the relation $E = \frac{1}{\sqrt{-2K}}$ which comes out to be $9.44 \pm 0.07\ kJ\ mol^{-1}$, with the value of $K = -5.60 \times 10^{-3}\ mol^2\ kJ^{-2}$. The obtained value and magnitude of E indicate that the adsorption presents a low potential

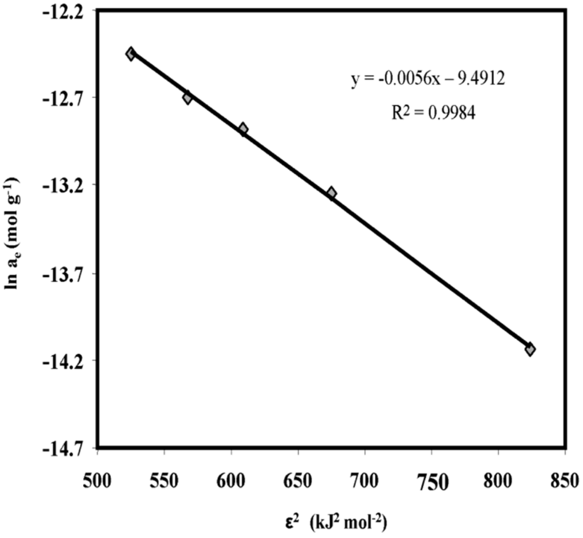

Figure 5. Dubinin-Radushkevich (D-R) isotherm of CrO_4^{2-} ions adsorption on Fe-modified pozzolane.

energy barrier. This value is more consistent of a physical adsorption process (23).

Effect of Temperature

The effect of temperature on the adsorption of CrO_4^{2-} ions on Fe-modified pozzolane was also investigated using the optimized conditions. The temperature was varied from 293 to 323 K. It was observed that the adsorption of CrO_4^{2-} ions increases with the increase in temperature (Table 1). The increase in amount of CrO_4^{2-} ions adsorbed with

Table 1. Temperature variation study on amount of adsorbed CrO_4^{2-} ions adsorbed on Fe-modified pozzolane at equilibrium: Initial concentration of adsorptive solution 1×10^{-4} mol L^{-1}; pH 5.5

	Amount of CrO_4^{2-} ions adsorbed	
Temperature (K)	(mol g^{-1})$\times10^{-6}$	Adsorption (%)
293	3.239 ± 0.01	32.39
303	3.414 ± 0.07	34.14
313	5.037 ± 0.02	50.37
333	6.424 ± 0.03	64.24

temperature suggests that the active, surface centers available for adsorption have increased with temperature.

Using the temperature dependence data, the thermodynamic parameters ΔH^0, ΔS^0, and ΔG^0 during the $CrO_4{}^{2-}$ ions adsorption process, have been evaluated utilizing the following equations:

$$\log K_d = -\frac{\Delta H^0}{2.303R}\left(\frac{1}{T}\right) + \frac{\Delta S^0}{2.303R} \tag{7}$$

$$\Delta G^0 = -RT\ ln\ K_d \tag{8}$$

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \tag{9}$$

where ΔH^0, ΔS^0, ΔG^0, and T are the change in standard enthalpy, change in standard entropy, change in Gibbs free energy and temperature in Kelvin, respectively, and R is the gas constant in kJ $mol^{-1}K^{-1}$. The change in enthalpy was calculated from the slope of straight line plot between log K_d and $\frac{1}{T}$ in Fig. 6, derived from the application of the van't Hoff equation in linearized form:

$$\log K_d = \frac{-\Delta H^0}{2.303RT} + constant \tag{10}$$

The value of change in enthalpy (ΔH^0) was found to be 49.87 ± 0.3 kJ mol^{-1} and the positive value, opposes the chromium adsorption which is

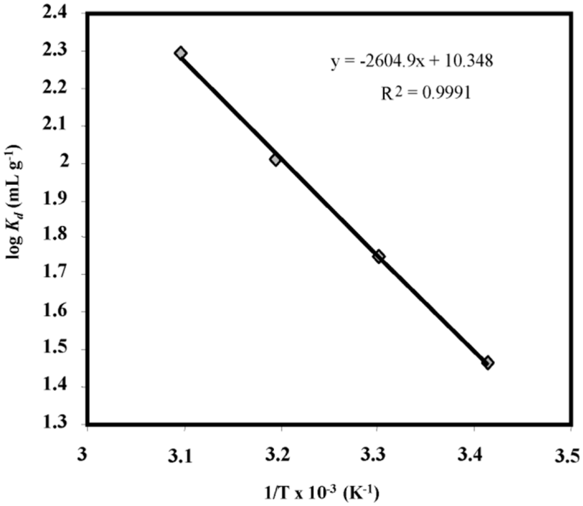

Figure 6. Plots of log *Kd* vs. *1/T* for the $CrO_4{}^{2-}$ ions adsorption on Fe-modified pozzolane.

due to a large enthalpy required for dehydration of the metal ion to be adsorbed on the Fe-modified pozzolane; additionally indicates the endothermic nature of adsorption (24). On the other hand, the value of change in entropy (ΔS^0) of the system, was also estimated to be $(19.81 \pm 0.2) \times 10^{-2}$ kJ $K^{-1}mol^{-1}$ and the positive value, indicates that the degree of freedom of ions is increased by adsorption. This is also an indication on the stability of surface adsorption. The value of change in Gibbs free energy ΔG^0 was found to be -8.17 ± 0.3 kJ mol^{-1}; the negative change in energy revels that the uptake process is spontaneous.

The described results indicate that the Fe-modified pozzolane is an adequate adsorbent for removal of chromate ions from aqueous solutions under the test experimental conditions. The benefits and novelty of using pozzolane-Fe instead of other solid studied and reported in the literature, for instance clinoptilolite modified with alkylammonium surfactants, activated alumina. activated charcoal, calcareous karst soils (18–20), include the abundance of pozzolane in nature, its low cost, and that it possesses great thermal stability, chemical resistance and low solubility in a wide pH interval.

CONCLUSIONS

The study showed the ability of Fe-modified pozzolane to adsorb CrO_4^{2-} ions from aqueous solution. When this material was prepared by adsorbing iron on its surface, it becomes an anionic exchanger and a considerable amount of CrO_4^{2-} ions from aqueous solution were retained. The adsorption was found to be initial concentration and temperature dependent in the equilibrium. Experimental isotherms of CrO_4^{2-} ions were adequately modeled by Freundlich, Langmuir, and Dubinin-Radushkevich (D-R) isotherms over the entire concentration range studied. The positive value of ΔH^0 indicates the endothermic nature of adsorption, the positive value of ΔS^0 indicates the increasing randomness of the system and ΔG^0 proves that the adsorption of CrO_4^{2-} ions on Fe-modified pozzolane is a spontaneous process. The Fe-modified pozzolane can be effectively used for the removal of CrO_4^{2-} ions from aqueous solutions and in the treatment processes of industrial wastes.

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