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**Decoration of *Ziziphus spina-christi* leaves derived-activated carbon with
Fe₃O₄ nanoparticles and SDS surfactant for removal of Cr³⁺ from polluted
water**

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Abstract

The purpose of this study was to remove Cr³⁺ ions from polluted water using AC@Fe₃O₄@SDS nanocomposite. The physical and surface features of the nanocomposite were investigated with EDS, DLS, XRD, BET, FTIR, SEM and TEM analyses. The BET surface area and the mean pore diameter of the AC@Fe₃O₄@SDS were 102.04 m²/g and 169.49 °A, respectively, indicating the high porosity of the aforementioned nanocomposite. DLS and XRD analyses showed that the mean particle size and mean crystal size of AC@Fe₃O₄@SDS were 78.9 nm and 28 nm, respectively. Also, the highest Cr³⁺ removal efficiency (99.5%) using the AC@Fe₃O₄@SDS nanocomposite was obtained at pH= 7, contact time= 40 min, temperature= 55 °C, the Cr³⁺ ion concentration= 10 mg/L and the nanocomposite dosage= 2 g/L, which is the highest Cr³⁺ removal efficiency ever achieved. Moreover, the utmost sorption capacity obtained from the Langmuir model was 120.48 mg/g, which is a significant value compared to previous studies for Cr³⁺ removal. Furthermore, the isotherm and kinetic studies of the sorption process indicated that the Cr³⁺ sorption process using the aforementioned nanocomposite followed the quasi-second order and Freundlich equations due to higher correlation coefficients. Subsequently, the thermodynamic study demonstrated that the sorption process of Cr³⁺ ions using AC@Fe₃O₄@SDS is favorable, feasible, spontaneous and endothermic.

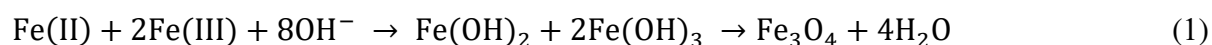
Keywords: AC@Fe₃O₄@SDS nanocomposite, Cr³⁺ ions, Polluted water, Sorption

1. Introduction

Water is the most important substance on earth that is used (Namdeo, 2017). The presence of heavy metal ion in aquatic solutions causes a plenty of problems for human and animal health. These contaminants are not biodegradable and cause different diseases. Chromium (Cr) is among the eleven most dangerous environmental contaminants (Tamjidi et al., 2021). Due to the high solubility of heavy metal ions in aquatic media, these metal ions may be adsorbed by living organisms (Barakat, 2010). Chromium ion is considered as a very toxic water pollutant. Chemicals industries, dyes, steelworks, leather tanning, photographic material, cement industries, and anti-corrosive paints generate high content of chromium wastes (Tamjidi et al., 2021). Though the trivalent chromium [Cr³⁺] is known to be less lethal than hexavalent chromium [Cr⁶⁺], its removal as a water-soluble species may significantly increase public health issues because it oxidizes to [Cr⁶⁺] in the natural environment. Exposure of excess chromium is expected to cause lung cancer and other respiratory diseases. Direct skin contacts with Cr³⁺ result in various issues like irritation of the skin (e.g., ulcers, rashes, and permanent scarring). Therefore, eliminating Cr³⁺ from wastewater, if it is excessive, is of great importance (Basu et al., 2019; Sun et al., 2022). Maximum allowed dosage of Cr³⁺ is considered 5 mg/L, according to WHO criteria (Almeida et al., 2019).

The most critical approaches for eliminating metal ions from sewages and aquatic solutions include ion exchange, chemical precipitation, membrane filtration, flocculation, electrochemical treatment and sorption (Yao et al., 2021). Among the above procedures, sorption by nanoparticles is environment friendly, effective, simple, affordable, without toxic and dangerous pollutants, conveniently generalized to large scales, highly reversible technology and it is a highly efficient technology for removing metal ions (Li et al., 2011).

Activated carbon (AC) is one of the most extensively utilized sorbents in water treatment due to its high sorption capacity and good stability. However, the biggest problem is its higher cost than other sorbents as well as the difficulty of recovering it (Tadda et al., 2016). Finding an alternative to AC, which is both abundant and inexpensive, is an issue that has received much attention. Lotus with scientific name of *Spinachristi Zizyphus*, is a plant from the family of jujube that grows wildly in Saudi Arabia, north Africa and southern provinces of Iran (Abshirini et al., 2019). Due to the abundance and low cost of this plant, in this study, its leaves were used to generate AC. Also, iron oxide nanoparticles were used to enhance the sorbent efficiency and its recoverability. Nano-adsorbents have many applications in water and wastewater treatment because of their high active surface area, aggregation properties, high reactivity and catalytic potential. In recent years, magnetite (Fe₃O₄) nanoparticles have been considered in the field of water treatment; because not only it is a strong sorbent but also an active regenerator. It is also easily separated from the environment by a strong magnetic field, which is useful in terms of recovery and reuse. Iron oxide comes in many forms in nature, with maghemite (γ-Fe₂O₃), magnetite (Fe₃O₄), and hematite (α-Fe₂O₃) being the most common. Fe₃O₄ nano-particles are the most investigated substances because of their superparamagnetic behaviors at 25 °C. In addition, they are non-toxic and highly bio-compatible (Petcharoen & Sirivat, 2012; Keshavarz et al., 2021). Furthermore, to prevent this nano-adsorbent from precipitation and agglomeration, its surface properties were improved with sodium dodecyl sulfate (SDS), which is one of the most powerful anionic surfactants (Sobhanardakani et al., 2016; Tamjidi and Esmaeili, 2019). The coprecipitation method maybe is the simplest and the most effective way to synthesize magnetic nanoparticles. In this procedure, Fe²⁺ and Fe³⁺ ions are stoichiometrically added to an aqueous solution and can produce superparamagnetic nanoparticles under alkaline conditions. The main reaction is occurred below:



Fe₃O₄ can be converted to γ -Fe₂O₃ in the presence of H⁺. Therefore, to prevent adverse reactions, it is necessary to control the reaction pH along the path. Large scale-production of nanoparticles as well as controlling their size are the most important advantages of the co-precipitation method. Fig. 1 illustrates the iron oxide production reaction (Li et al., 2011).

Figure 1

In this study, the AC@Fe₃O₄@SDS nanocomposite was used as a fantastic sorbent to eliminate Cr³⁺ from synthetic wastewater. The surface characterizations of AC@Fe₃O₄@SDS were investigated by SEM, TEM, EDS, FTIR, DLS, XRD and BET analyses. Also, the impact of temperature, pH, contact time, Cr³⁺ ion concentrations and nanocomposite dosage on Cr³⁺ sorption was examined and the best sorption efficiency was obtained. Moreover, kinetic, equilibrium, and thermodynamic models were employed to survey the sorption behavior of the nanocomposite. To the best of our knowledge, AC@Fe₃O₄@SDS was produced and used for the first time in this work.

2. Materials and procedures

2.1. Chemicals and devices

FeCl₂.4H₂O, FeCl₃.6H₂O (Merck Company, purities>0.99) and sodium dodecyl sulfate (SDS, Merck, purity=0.98) were used to synthesize AC@Fe₃O₄@SDS nanocomposite. Also, chromium trichloride hexahydrate (CrCl₃.6H₂O, Merck, purity=0.99) was employed to prepare chromium stock solutions. NaOH and HCl (Merck Company, purities>0.99) were also used to regulate pH using pH meter (Metrohm 44 model).

A flame atomic absorption device (Younglin AAS8020 model) was used to specify the remaining chromium ions in solutions. Also, a digital scale (Sartorius model) and magnetic-stirrer (HS-860) were used to measure the samples weight and stir the solutions, respectively.

Moreover, an oven (ATBIN MEGA model) was used to dry the AC@Fe₃O₄@SDS nanocomposite.

2.2. Chromium stock solutions

To prepare chromium (III) ion stock solutions with primary concentration of 1000 ppm, 5.12 g of CrCl₃.6H₂O salt was added to double distilled water. Then, for synthesizing stock solutions with lower concentrations, the primary stock solution was diluted.

3.3. Preparing AC@Fe₃O₄@SDS nanocomposite

AC@Fe₃O₄@SDS nanocomposite was produced by chemical coprecipitation method. First, lotus leaves were gathered and washed several times to wipe out any dust and impurities. The leaves were then placed in an oven at 105°C for 2 hrs for drying. Next, they were turned into charcoal at 450 °C for 4 h. After that, the charcoal was poured into a 3 M NaOH solution and stirred with a magnetic-stirrer for 2 h. Then, the charcoal was isolated from the suspension and dried at 105°C for 2 hrs. Finally, the charcoal was placed in the furnace at 700 °C for 4 to be fully activated.

To prepare AC@Fe₃O₄@SDS nanocomposite, a solution containing Fe³⁺ and Fe²⁺ (molar ratio of 2:1) was prepared. Then, 1 g of activated carbon powder and 0.5 g of SDS were added to the solution at 60 °C and the suspension was stirred for 20 min at 400 rpm. Next, 3 M NaOH solution was added dropwise to the mix to make the solution black. After that, the synthesized nanocomposite was isolated from the suspension by a N42 magnet and washed with distilled water to neutralize. Finally, the AC@Fe₃O₄@SDS nanocomposite was put in the oven at 105

°C for a day to completely dry. Fig. 2 shows a schematic of the synthesis of the AC@Fe₃O₄@SDS nanocomposite and its use for Cr³⁺ removal.

Figure 2

To analyze the sorption features of AC@Fe₃O₄@SDS nanocomposite, SEM, EDS (TESCAN, MIRA III model, Czech Republic), BET (ASAP2020, MICROMERITICS, USA), XRD (PHILIPS, PW1730, Netherlands), DLS (HORIBA, SX100, Japan), FTIR (THERMO, AVATAR, USA), EDS, and TEM (PHILIPS, CM120, Netherlands) analyses were done.

3.4. Sorption Experiments

Sorption experiments were discontinuously done into 250 mL Erlenmeyer flasks containing 50 mL of chromium ions using AC@Fe₃O₄@SDS nanocomposite. To this end, impact of various factors in several levels was studied on the sorption process, including pH (3-11), temperature (25-55 °C), time (10-120 min), chromium (III) ion concentration (5-50 mg/l) and nanocomposite dose (0.25-5 g/L). The rate of mixing for all experiments was considered 200 rpm. After each experiment, the residual concentration of Cr³⁺ ions in the suspension was specified by atomic absorption device. For investigating the impact of pH, several experiments were done at various pHs (3, 5, 7, 9 and 11) while other factors were kept constant (T=25 °C, time= 80 min, adsorbent dose=2 g/L and Cr³⁺ concentration= 10 ppm). After performing the experiments, the best pH value was determined at the highest Cr (III) removal efficiency. Sorption efficiency (R%) and sorption capacity (q_e) of AC@Fe₃O₄@SDS nanocomposite can be calculated as follows:

$$\%R = \frac{(C_i - C_o)}{C_i} \times 100 \quad (2)$$

$$q_e = \frac{C_i - C_o}{M} \times V \quad (3)$$

where, C_i (mg/l), C_o (mg/l), M (g) and V (L) are initial concentration, Cr^{3+} equilibrium concentration, the nanocomposite mass and the Cr^{3+} solution volume, respectively.

Sorption isotherms

Sorption isotherm equations are utilized to describe the equilibrium behavior of adsorbed molecules on the sorbent surface. Freundlich and Langmuir isotherms are the most critical equations applied in the uptake process. The Langmuir isotherm assumes that the uptake of contaminants occurs as a mono-layer on the nanocomposite surface and there is no interaction between the sorbent and the adsorbed component (Eq. 4) (Esmaeili and Tamjidi, 2020):

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{q_{max}K_L} \quad (4)$$

where C_e , q_e , K_L , and q_{max} are the equilibrium concentration of the adsorbed adsorbate (mg/L), the amount of adsorbed adsorbate per gram of the nanocomposite (mg/g), the Langmuir constant, and the utmost sorption capacity (mg/g), respectively (Mohammed et al., 2019). Also, the Freundlich model assumes that the uptake of pollutants takes place on the heterogeneous surfaces of the sorbent and in multiple layers (Eq. 5) (Ahmed and Hameed, 2019):

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (5)$$

where n and K_f are the Freundlich model constants. Also, n describes the deviation of the sorption process from linearity. For $1 < n < 10$ the uptake process is physical and desirable (Velooso et al., 2020).

Sorption kinetics

A powerful way to understand the reaction mechanism is to check the sorption kinetics. It is a critical factor in evaluating the efficiency, design and modeling of the adsorption process (Mohammed et al., 2019). The most commonly used kinetic models are quasi-first-order (QFO)

and quasi-second-order (QSO) kinetic models as defined in Eqs. 6 & 7 (Esmaeili and Tamjidi, 2020).

$$\ln(q_e - q_t) = \ln(q_e) - k_1 t \quad (6)$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad (7)$$

where q_t , k_1 , and K_2 are the amount of adsorbed ion per gram of sorbent at time t (mg/g), the constant sorption rate of the QFO model (1/min) and the equilibrium constant of the QSO model (g/mg.min), respectively (Inyinbor et al., 2016).

Sorption thermodynamic

Thermodynamic parameters are essential to specify the spontaneity of a sorption process. Thermodynamic indices for the sorption process are assessed by calculating Gibbs standard free energy variations (ΔG°), standard enthalpy variations (ΔH°) and standard entropy variations (ΔS°), as described below:

$$-RT \ln(55.5 K_D) = \Delta G^\circ \quad (8)$$

$$\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ \quad (9)$$

where R and K_D are the universal gas constant ($8.314 \text{ J mol}^{-1} \cdot \text{K}^{-1}$) and the equilibrium constant obtained from Eq. 10, respectively. Also, the number 55.5 represents the amount of water moles per solution liter.

$$K_D = \frac{q_e}{C_e} \quad (10)$$

A negative value of ΔG° demonstrates the spontaneous nature of the uptake process. Absolute values between -20 to 0 indicate physical sorption and between -80 to -400 kJ/mol means chemical sorption (Foroutan et al., 2019).

$$\ln(55.5 K_D) = \frac{-\Delta G^\circ}{RT} = \frac{-\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \quad (11)$$

A positive value of ΔH° indicates that the uptake process is endothermic, while its negative value demonstrates that the pollutant uptake process is exothermic. Positive values of ΔS° show an increase in the solid-solution surface irregularity during the uptake process, which causes slight structural changes in the sorbent and the adsorbed component; on the other hand, negative values of ΔS° indicate a reduction in irregularity in the uptake process.

3. Results and discussion

3.1. Characterization of AC@Fe₃O₄@SDS

Functional groups in the structures of AC and AC@Fe₃O₄@SDS before and after uptake of Cr³⁺ ions were determined by FTIR analysis and outputs are illustrated in Fig. 3. In AC, an absorption peak was observed at 454.5 cm⁻¹, which is attributed to Ca-O bond. 2 peaks were also seen at 572.74 and 604.74 cm⁻¹, which are related to Fe-O bond. The next peak was appeared at 876.05 cm⁻¹ related to C-H aromatic group. Two other peaks at 956.49 and 1050.94 cm⁻¹ belong to the R₃N-O aliphatic and C-OH phenol groups, respectively. Also, two peaks at 1251.93 and 1461.36 cm⁻¹ belong to the O-C-O carbonate group and the two other peaks at 1642.14 and 1725.51 cm⁻¹ belong to the C=O tensile vibration. Finally, -COOH vibration was seen at 2925.65 cm⁻¹ as well as N-H amide vibration was seen at 3432.75 and 3643.57 cm⁻¹. Fig. 3 (b) shows the FTIR spectra of AC@Fe₃O₄@SDS nanocomposite before adsorption. The appeared peak at 466.87 cm⁻¹ belongs to the Ca-O tensile vibration. Two absorption peaks at 704.06 and 822.08 cm⁻¹ belong to the C-H bond. Also, two peaks at 1056.51 and 1252.21 cm⁻¹ are attributed to the O-C-O carbonate and S=O tensile groups, respectively. Another peak at 1386.96 cm⁻¹ belongs to the asymmetric vibration bond of S=O in sulfonic acid. Moreover, a peak was seen at 1462.11 cm⁻¹, which is attributed to the O-C-O carbonyl group. Furthermore, the C=O tensile vibration is at 1633.47 and 1727.98 cm⁻¹. Also, two other peaks at 2852.14 and 2923.31 cm⁻¹ belong to the -COOH functional group and peak at 3427.54 cm⁻¹ is related to the

O-H hydroxyl vibration (Due to the absorption of water in the nanocomposite surface). Fig. 3(c) indicates the FTIR spectra of AC@Fe₃O₄@SDS nanocomposite after adsorption of Cr³⁺. Peak at 468.86 cm⁻¹ belongs to the Ca-O tensile vibration. Also, C-H aromatic bond was appeared at 699.64 cm⁻¹. Moreover, two absorption peaks were seen at 1059.58 and 1246.84 cm⁻¹, which are attributed to the O-C-O carbonate group and S=O vibration absorption in SDS, respectively. The appeared peak at 1462.96 cm⁻¹ is also related to the O-C-O carbonate group. The C=O tensile vibration is appeared at 1632.06 cm⁻¹. Furthermore, absorption peaks at 2853.09 and 2922.67 cm⁻¹ belong to the -COOH carboxyl group and peak at 3425.91 cm⁻¹ belongs to the O-H hydroxyl group vibration (Gupta et al., 2011; Silva et al., 2013; Burks et al., 2014).

Figure 3

Also, for characterizing the surface morphology of AC and AC@Fe₃O₄@SDS nanocomposite before uptake of chromium (III) ions, SEM analysis was employed and outputs are demonstrated in Fig. 4. As indicated in Fig. 4(a), AC particles have a spherical and porous structure, indicating the suitability of the synthesis of AC@Fe₃O₄@SDS nanocomposite. Fig. 4(b) shows that before the sorption process, there are many bumps and pores in the AC@Fe₃O₄@SDS structure so that Fe₃O₄ nanoparticles are placed inside the AC layers and then SDS improves its surface, leading to unevenness and enhances the nanocomposite specific surface area. Also, after uptake of Cr³⁺ as shown in Fig. 4(c), many pores and holes in the AC@Fe₃O₄@SDS surface are well covered by Cr³⁺ ion.

Figure 4

Crystalline phases in the AC and AC@Fe₃O₄@SDS nanocomposite structures were specified by XRD spectra. X-pert software was employed to draw XRD patterns and the outcomes are illustrated in Fig. 5. Several compounds such as Ca(OH)₂, Ca₁₀(OH)₂(PO₄)₆, periclase

(Mg₁O₁), and alite (Ca₃O₅Si₁) were identified in the AC structure, which are attributed to card numbers of 01-076-0570, 00-001-1008, 98-006-2127 and 98-011-5657, respectively (Pontremoli et al., 2018; Zhang et al., 2020). Also, several peaks were seen in the AC@Fe₃O₄@SDS nanocomposite structure at 27, 35.5 and 63.5°, which correspond to the card number 98-001-7122 (Faiyas et al., 2010).

Figure 5

The mean size of crystals was specified via $d = \frac{K \lambda}{\beta \cos \theta}$ (Debye-Scherrer model). In this model, d , λ , β and θ are the crystal size (nm), the wavelength of the apparatus (0.15406 nm), Full Width at Half Maximum, and tilt angle, respectively. Also, Scherrer constant (K) is considered as 0.9 (Gupta et al., 2011). The mean size of crystals for AC and AC@Fe₃O₄@SDS were obtained 217 and 28 nm, respectively.

Surface features and volume of pores for AC and AC@Fe₃O₄@SDS were specified using BET analysis (Fig. 6). The BET surface area, Langmuir surface area, the mean diameter of pores and total volume of pores for AC were 13.52 m²/g, 13.39 m²/g, 467.78 °A, and 0.158 cm³/g, respectively. Also, these features for AC@Fe₃O₄@SDS nanocomposite were 102.04 m²/g, 94.31 m²/g, 169.49 °A, and 0.432 cm³/g, respectively.

Figure 6

Moreover, elemental compositions in the AC@Fe₃O₄@SDS structure before and after uptake of Cr³⁺ were determined by EDS analysis (Fig. 7). Also, the percentage of elements are reported in Table 1. As shown, in the AC@Fe₃O₄@SDS structure, the weight percentage of C, O and Fe were 13.56, 43.27 and 20.54%, respectively. Also, the weight percentage of other elements such as Na, P, S, Ca and Mg were 2.04, 5.42, 6.11, 3.73 and 5.33%, respectively. After adsorption of Cr³⁺ using the nanocomposite, the element percentage was changed slightly. For

example, the weight percentage of C, O and Na were 18.69, 35.05 and 0.09%, respectively. Chromium also appeared with 0.16% after adsorption.

Figure 7

Table 1

Furthermore, DLS analysis was utilized to specify the particle size distribution and measure the particle size of AC and AC@Fe₃O₄@SDS composite. The outcomes are illustrated in Fig. 8(a and b). As shown, the average particle size of AC and AC@Fe₃O₄@SDS were obtained 81.9 and 78.9 nm, respectively. Finally, TEM analysis was employed to specify the size and morphology of AC@Fe₃O₄@SDS. Fig. 8(c) shows that the size of particles is less than 100 nm.

Figure 8

3.2. Sorption results

The initial pH of the solution has impressive impacts on the sorption process. The solution pH not only affects the surface charge of the sorbent, but also affects its ionization degree (Abdel-Halim & Al-Deyab et al., 2011). The impact of pH (3-11) was studied on uptake of Cr³⁺ from polluted water using the AC@Fe₃O₄@SDS nanocomposite at constant conditions like t=80 min, Cr³⁺ concentration=10 mg/L, T=25 °C, the nanocomposite dosage= 2 g/L and mixing rate=200 rpm (Fig. 9(a)). At acidic pHs, the predominant forms of chromium ions are Cr³⁺, CrOH²⁺ and CrOH₂⁺. Under acidic conditions, the sorbent surface becomes protonated and as a result, the electrostatic attraction between chromium (III) species and the nanocomposite surface reduces, ultimately leading to a decrease in the sorption percentage. However, the concentration of protons on the nanocomposite surface reduces with increasing pH and will creates a strong attraction with cationic species of chromium (III). Chromium ions at alkaline pHs are as CrO₄²⁻, Cr₂O₄²⁻ and HCrO₄⁻, while at neutral pH, its forms will be CrO₄²⁻, Cr₂O₇²⁻

and HCrO_4^- (Bedemo et al., 2016). The outcomes indicated that the chromium sorption efficiency enhances with enhancing pH from 3 to 7 and the maximum sorption efficiency was attained at 97.98%. By raising pH from 7 to 9, no significant change in sorption efficiency was observed. Increasing the chromium sorption efficiency with pH can be due to the low concentration of H^+ compared to chromium ions for placement on the nanocomposite active sites. At higher pHs (9-11), the OH^- concentration in the suspension is high, and these ions compete with chromium ions for placement on the nanocomposite surface, thereby reducing the sorption efficiency (Baijnath et al., 2014).

Figure 9

Temperature has an impressive impact on sorption because it determines whether the sorption process is endothermic and/or exothermic. The effect of temperature in the range of 25-55 °C was studied on the Cr^{3+} sorption efficiency by $\text{AC@Fe}_3\text{O}_4\text{@SDS}$ (Fig. 9(b)). To investigate the impact of temperature, other factors like pH (7), chromium (III) concentration (10 ppm), the composite dose (2 g/L) and time (80 min) were kept constant. From Fig. 9(b), the Cr^{3+} sorption efficiency increases with raising temperature so that the maximum sorption efficiency (99.5%) occurs at 55 °C, indicating that the Cr^{3+} sorption efficiency by $\text{AC@Fe}_3\text{O}_4\text{@SDS}$ is endothermic.

Also, the impact of time (10-120 min) on the Cr^{3+} sorption efficiency by $\text{AC@Fe}_3\text{O}_4\text{@SDS}$ was studied at pH 7, temperature of 55 °C, the composite dose of 2 g/L and Cr^{3+} concentration of 10 ppm (Fig. 9(c)). The outcomes indicate that the sorption efficiency of Cr^{3+} ions increases with raising time, and the sorption process includes two steps. In the first step, the Cr^{3+} sorption process occurred very quickly due to the high active sites of the nanocomposite. In this step, the Cr^{3+} sorption efficiency reached 99.18% after 40 min. However, the second step was very slow and the Cr^{3+} sorption efficiency increased from 99.18 to 99.74% with enhancing time from 40 to 120 min, respectively. Hence, 40 min was considered as the optimal time. In fact,

the active sites of the nanocomposite were gradually occupied and the sorption percentage slowly decreased (Baijnath et al., 2014; Bedemo et al., 2016).

Fig. 9(d) shows the impact of Cr^{3+} ions concentration on the sorption efficiency and sorption capacity using $\text{AC@Fe}_3\text{O}_4\text{@SDS}$. Other factors like pH (7), contact time (40 min), temperature (55 °C), mixing speed (200 rpm) and the nanocomposite dose (2 g/L) were considered constant. As shown, the sorption capacity increases and the sorption efficiency decreases with enhancing the Cr^{3+} ion concentration. At low Cr^{3+} concentrations, there are many active sites on the nanocomposite surface that are sufficient to grab all the chromium ions. But the number of Cr^{3+} molecules increases at high concentrations and all of them can not be located on the active sites of the nanocomposite, thus reducing the sorption efficiency (Panda et al., 2017). Therefore, the maximum sorption efficiency (99.5%) was achieved at a concentration of 10 mg/L Cr^{3+} .

Adsorbent dosage is one of the most critical factors on the sorption process because it determines the capacity of a sorbent to adsorb contaminants (Ghnimi & Srasra, 2019). Fig. 9(e) shows the impact of sorbent dosage (0.25-5 g/L) on uptake of Cr^{3+} using the $\text{AC@Fe}_3\text{O}_4\text{@SDS}$ nanocomposite at optimized conditions (pH=7, temperature= 55 °C, time=40 min, Cr^{3+} concentration= 10 mg/L and mixing speed= 200 rpm). As demonstrated, the removal efficiency of Cr^{3+} increases with raising the nanocomposite dose from 0.25 to 2 g/L and at the nanocomposite dose higher than 2 g/L, the removal efficiency remained constant. Also, the sorption capacity decreased with raising the nanocomposite dose. Increasing the sorbent dosage increases the nanocomposite active sites to adsorb Cr^{3+} . As a result, the diffusion of Cr^{3+} ions into the nanocomposite active sites is facilitated and the sorption efficiency is increased (Panda et al., 2017). On the other hand, increasing the nanocomposite active sites reduces the Cr^{3+} sorption capacity and the maximum sorption capacity was obtained at a lower sorbent dose. Eventually, the highest sorption efficiency (99.5%) took place at a composite dose of 2 g/L as well as the highest sorption capacity (37.56 mg/g) took place at a composite dose of 0.25 g/L.

3.3. Sorption isotherms of Cr³⁺ ions using AC@Fe₃O₄@SDS

Sorption isotherm models describe the sorption interaction between sorbate (Cr³⁺) and sorbent (AC@Fe₃O₄@SDS) in equilibrium state (Esmaeili and Tamjidi, 2020). To study the sorption isotherms, several tests were done in different dosages of the nanocomposite (0.25-5 g/L). Other parameters were in optimized conditions (pH=7, Cr³⁺ ion concentration=10 mg/L, time=40 min, temperature= 55 °C and mixing speed= 200 rpm). The behavior of the uptake isotherms was investigated by two known models (Langmuir and Freundlich) and outputs are reported in Table 2 and illustrated in Fig. 10. As reported, the dimensionless parameter RL was obtained 0.123, demonstrating that the Cr³⁺ ions sorption process with the aforementioned nanocomposite is desirable and reversible (Mohammed et al., 2019). Also, the Freundlich constant (n) was obtained at 1.23, indicating that the uptake process is physical. Moreover, the utmost sorption capacity by the Langmuir model for uptake of Cr³⁺ using the AC@Fe₃O₄@SDS nanocomposite was 120.48 mg/g, which is a remarkable amount. Finally, the correlation coefficient (R²) indicates that the Freundlich isotherm is more proper than the Langmuir isotherm to describe the Cr³⁺ sorption process by AC@Fe₃O₄@SDS due to its higher R².

Figure 10

Table 2

The results of this study for removing Cr³⁺ ions were compared with previous studies. As shown in Table 3, AC@Fe₃O₄@SDS has a significant sorption capacity compared to other sorbents and can be employed as a promising sorbent for industrial wastewater treatment.

Table 3

3.4. Sorption kinetics of Cr³⁺ ion using AC@Fe₃O₄@SDS

For investigating the kinetic behavior of Cr^{3+} sorption, several analyses were done at the optimal pH (7), Cr^{3+} concentration of 10 ppm, temperature of 55 °C, a mixing speed of 200 rpm and different times (10-120 min). To do so, the QFO and QSO kinetic models were utilized for fitting the laboratory data (Table 4 and Fig. 11) (Zhao et al., 2016). As reported, the value of R^2 for QFO and QSO models is 0.976 and 1, respectively, describing the higher ability of the QSO model than QFO model to adsorb Cr^{3+} using the $\text{AC@Fe}_3\text{O}_4\text{@SDS}$ nanocomposite. The q_e calculated from the QSO model is more consistent with the laboratory data than the QFO model.

Figure 11

Table 4

3.5. Thermodynamic investigation

To investigate the thermodynamic behavior of the uptake process, several tests were performed at different temperatures (25-55°C). Other factors were kept constant (pH=7, t=40 min, the composite dose=2 g/L, Cr^{3+} concentration=10 ppm and mixing speed=200 rpm). Fig. 12 and Table 5 present the constants and parameters of the thermodynamic study. As reported, the amount of ΔG° for all temperatures became negative and their values are large, indicating that the Cr^{3+} sorption process using the $\text{AC@Fe}_3\text{O}_4\text{@SDS}$ nanocomposite is spontaneous and physical. Also, the value of ΔH° is positive, showing the endothermic nature of the Cr^{3+} sorption process using the $\text{AC@Fe}_3\text{O}_4\text{@SDS}$ nanocomposite. On the other hands, the uptake process increases with raising temperature (Singh et al., 2018; Al-Senani & Al-Fawzan, 2018). Moreover, the amount of ΔS° is positive for the Cr^{3+} sorption process using the $\text{AC@Fe}_3\text{O}_4\text{@SDS}$ nanocomposite, indicating an increase in randomness at the solid-solution interface is expected during the uptake process (Singh et al., 2018; Yegane Badi et al., 2018).

Figure 12

Table 5

4. Conclusion

In this work, the adsorption of chromium(III) from contaminated water was studied using the AC@Fe₃O₄@SDS nanocomposite. To prepare AC, lotus leaves were utilized and the nanocomposite was synthesized by the coprecipitation process. Several analyses like TEM, EDS, DLS, XRD, BET, FTIR and FESEM were employed to study the structural features of the aforementioned sorbent. Different factors like pH (3-11), temperature (25-55°C), time (10-120 min), Cr³⁺ ions concentration (5-50 ppm) and the nanocomposite concentration (0.25-5 g/L) were surveyed on the removal of Cr³⁺ ions from water with AC@Fe₃O₄@SDS. According to our results, the highest sorption efficiency of Cr³⁺ ions was 99.5%, which was attained at pH 7, temperature of 55 °C, Cr³⁺ concentration of 10 ppm, the AC@Fe₃O₄@SDS dosage of 2 g/L and time of 40 min. This is the highest removal efficiency of Cr³⁺ ion ever achieved. Also, the isotherm and kinetic studies exhibited that the Freundlich isotherm and PSO kinetic models were able better for describing the sorption of Cr³⁺ ions using the AC@Fe₃O₄@SDS nanocomposite because these two models had higher correlation coefficients than other isotherm and kinetic models. Moreover, thermodynamics of the sorption process confirmed that sorption of Cr³⁺ ions using the AC@Fe₃O₄@SDS nanocomposite is endothermic (enthalpy change>0), favorable and spontaneous (Gibbs free energy changes<0).

Competing Interests

Not applicable.

Declaration statement

Not applicable.

408 **Ethical Approval**

409 No approval was required. This manuscript is the authors' own original work, which has not
410 been previously published elsewhere.

411 **Consent to Participate**

412 All authors contributed to this work.

413 **Consent to Publish**

414 All authors agree to publish.

415 **Authors Contributions**

416 Shima Shiraghaei Koutenaei: Conceptualization, data curation, methodology, software.

417 Gholamhossein Vatankhah: Conceptualization, methodology, supervision, project
418 administration.

419 Hossein Esmaeili: Conceptualization, Data curation and methodology. Writing-review and
420 editing, Validation.

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423 **Availability of data and materials**

424 Not applicable.

425

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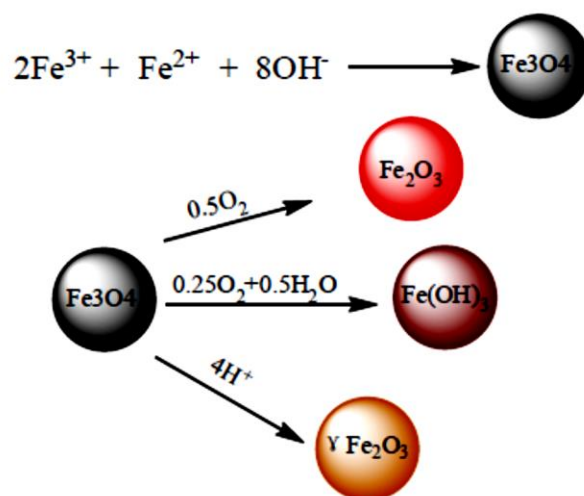
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540 **Figure captions:**



541

542 Figure 1. The production reaction of iron oxide

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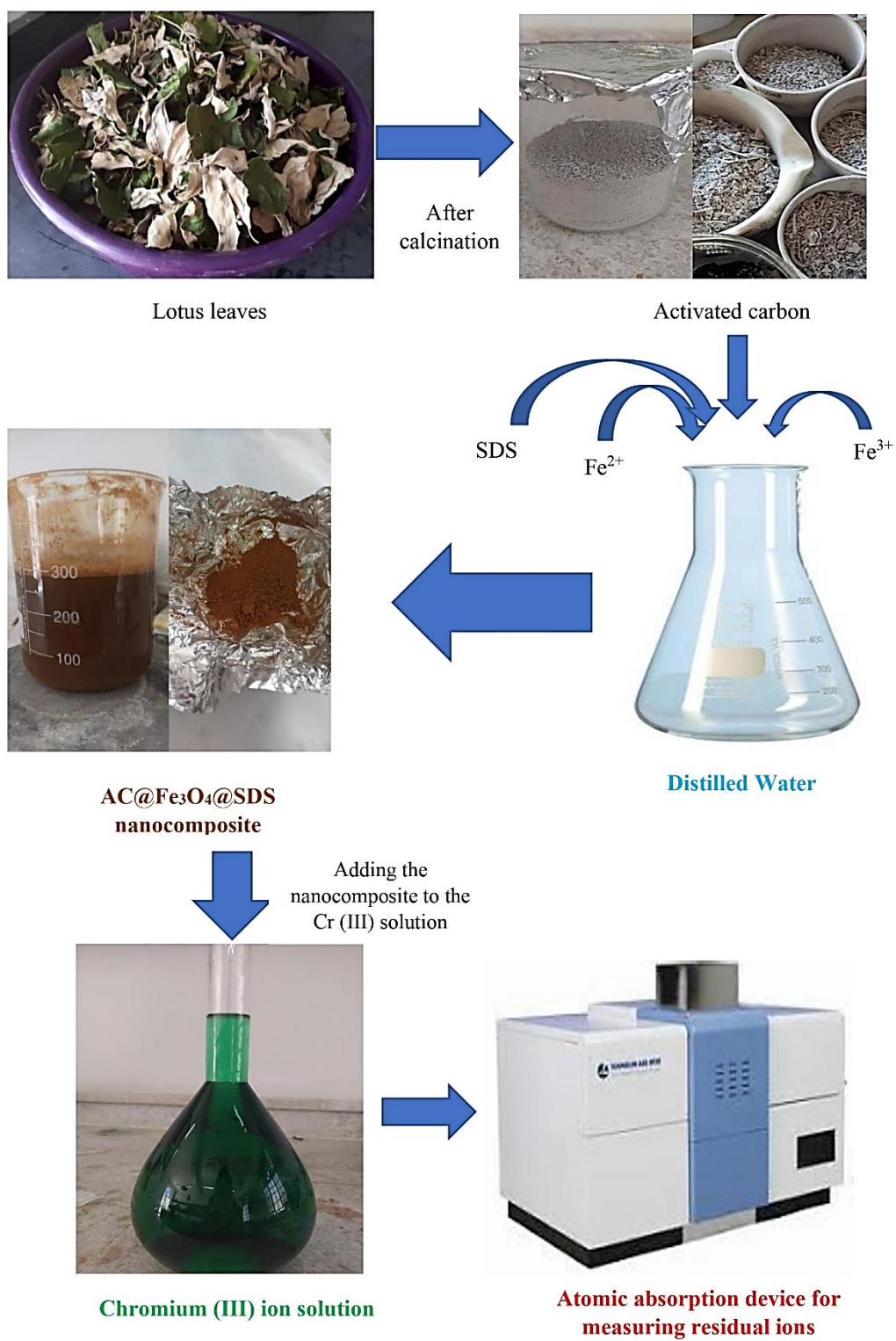
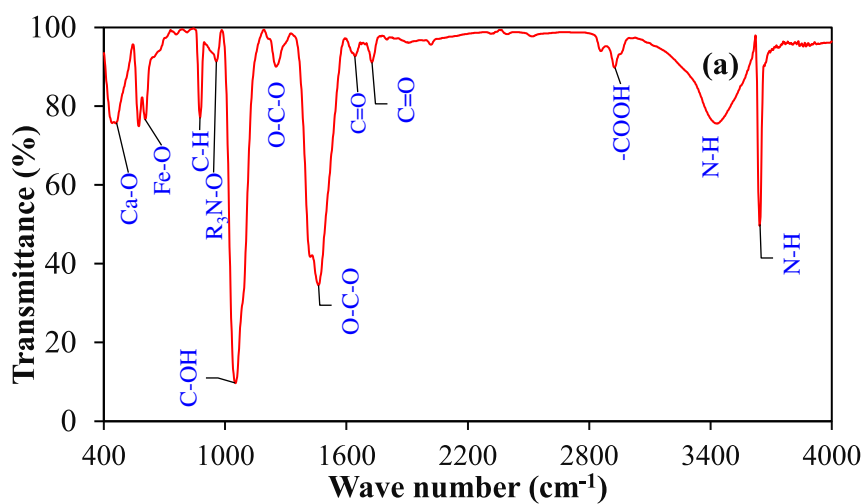
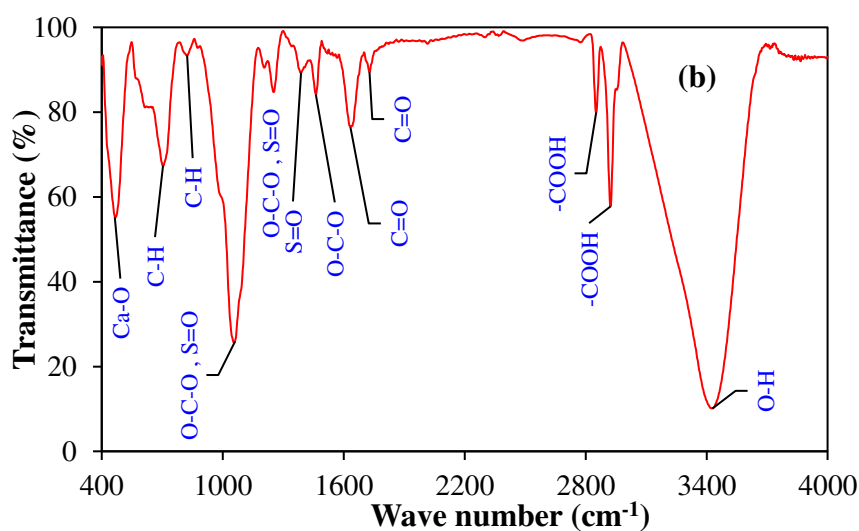


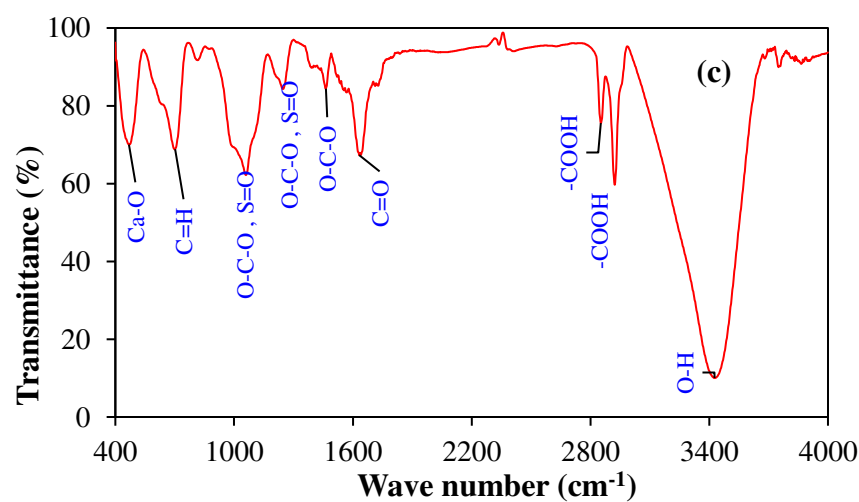
Figure 2. Synthesis of AC@Fe₃O₄@SDS and its application in removing Cr³⁺ ions from polluted water



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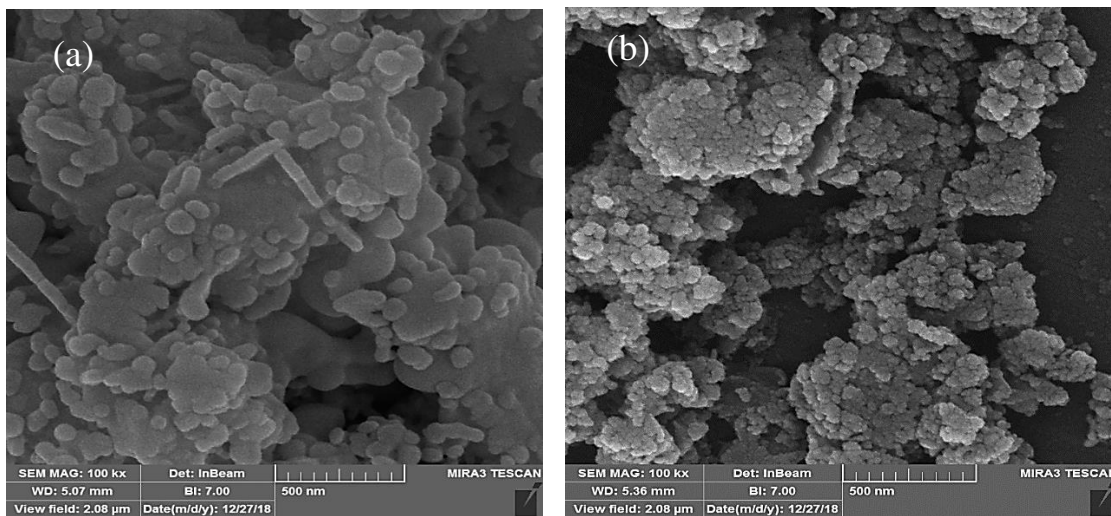


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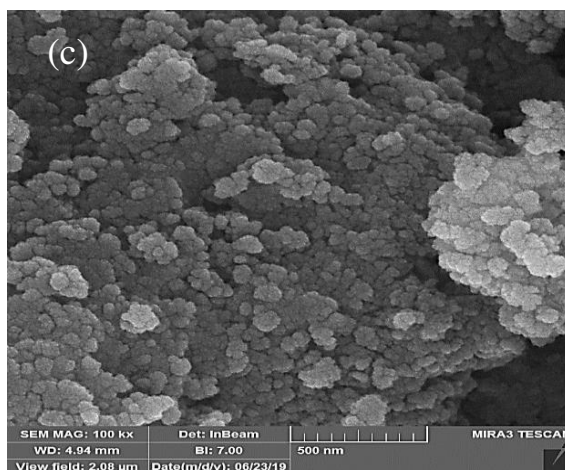
551 Figure 3. FTIR analysis for AC (a), AC@Fe₃O₄@SDS nanocomposite before uptake of
552 chromium (b) and after uptake of chromium ions (c)

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556 Figure 4. SEM images of AC (a), AC@Fe₃O₄@SDS (b) and AC@Fe₃O₄@SDS+Cr³⁺ (c)

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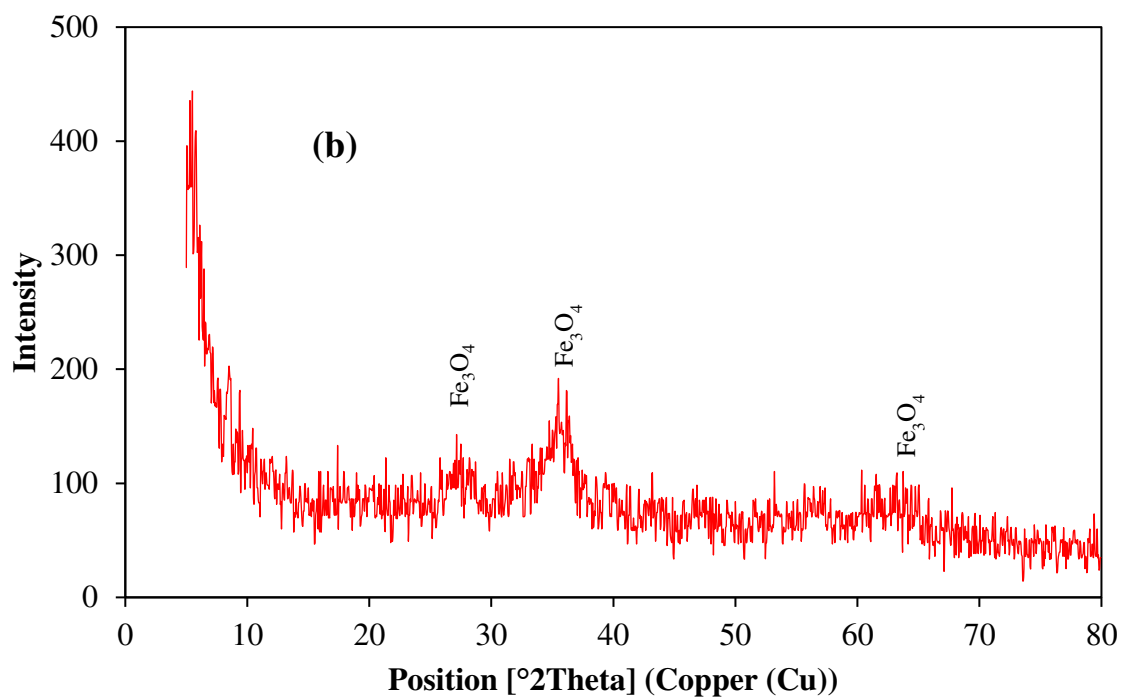
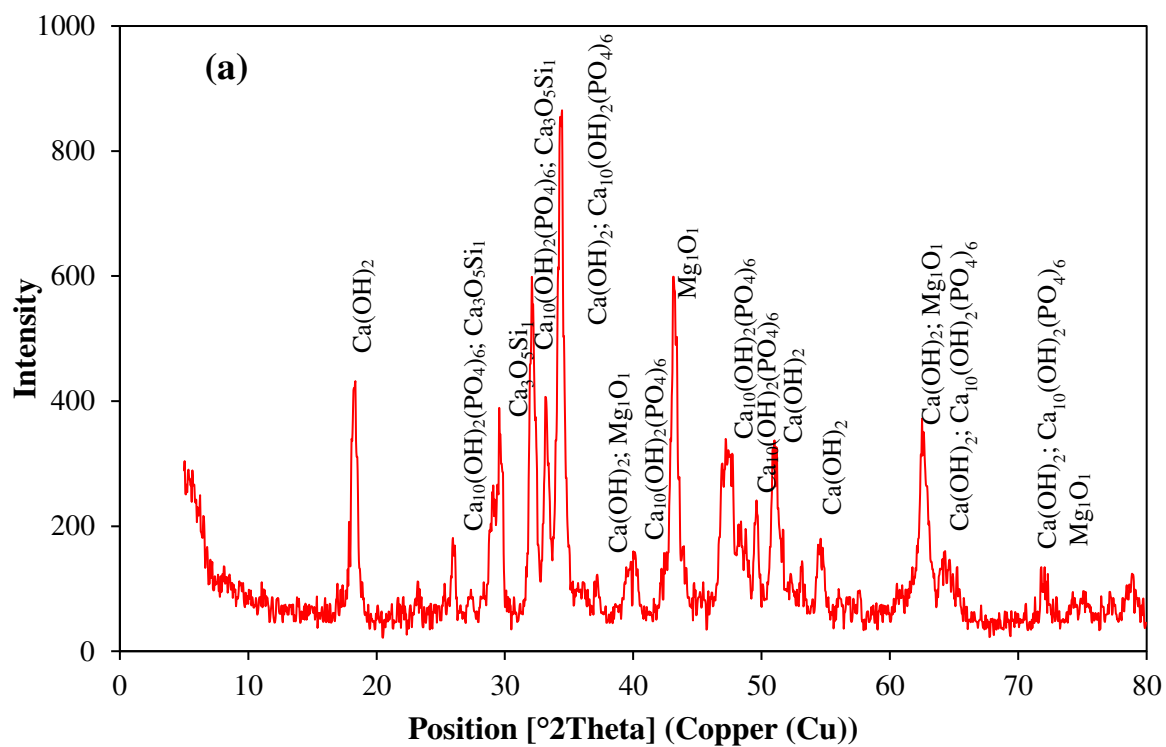


Figure 5. XRD patterns for AC and AC@Fe₃O₄@SDS nanocomposite

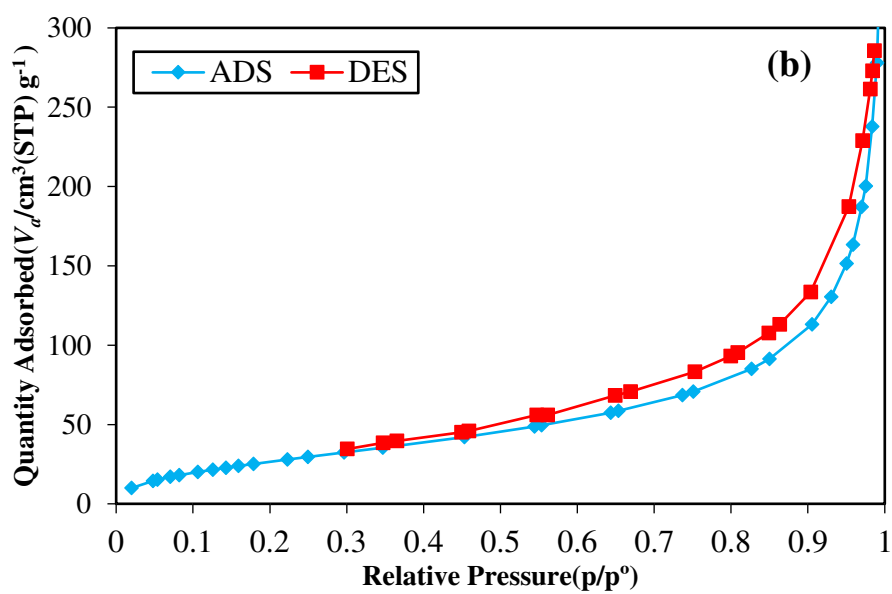
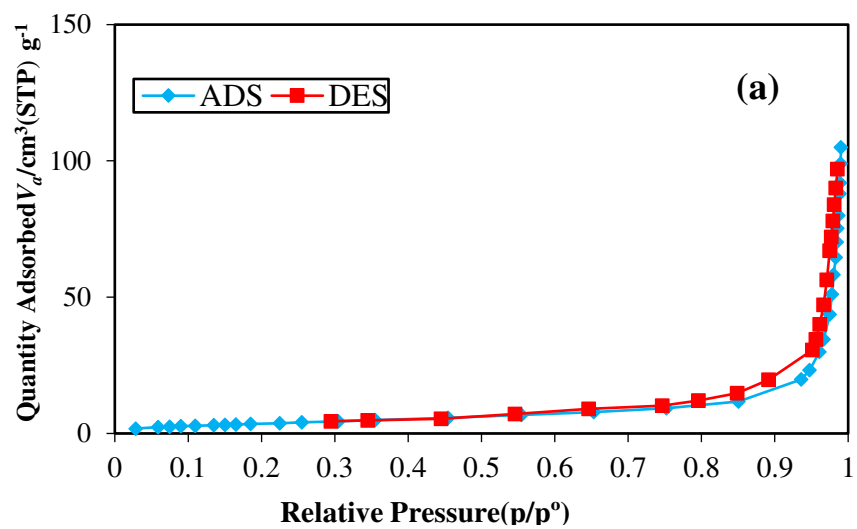


Figure 6. BET analyses for AC (a) and AC@Fe₃O₄@SDS (b)

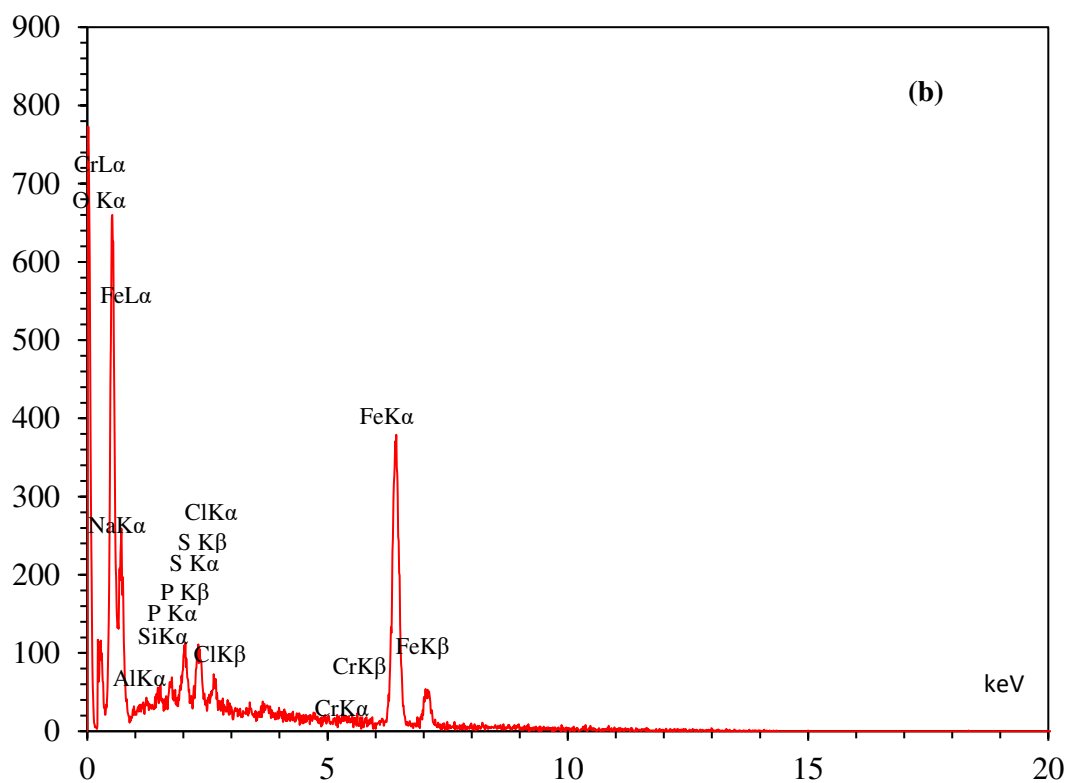
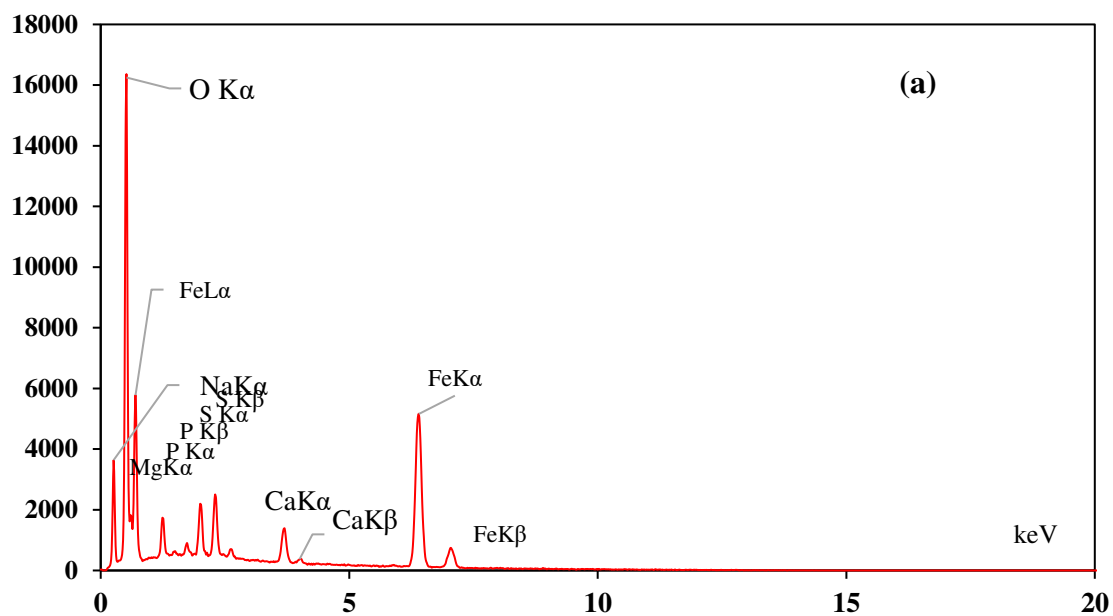


Figure 7. EDS analyses for AC@Fe₃O₄@SDS (a) and AC@Fe₃O₄@SDS+Cr³⁺ (b)

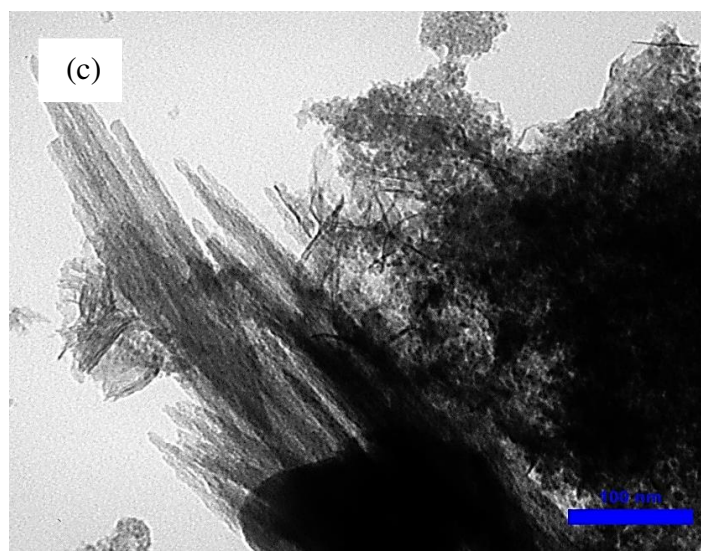
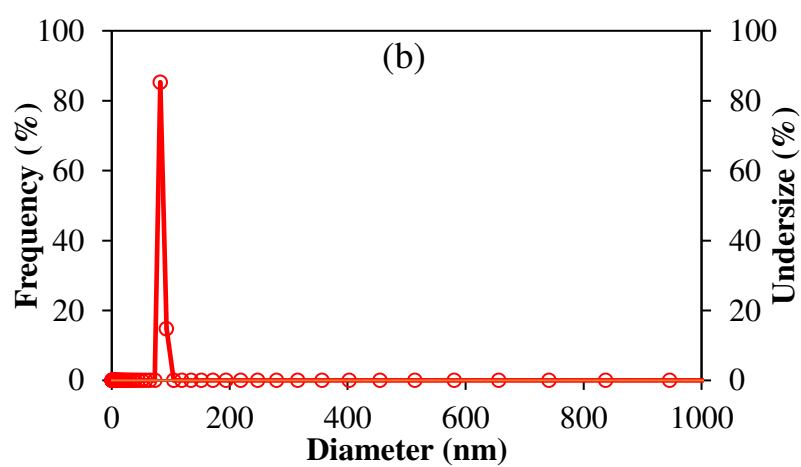
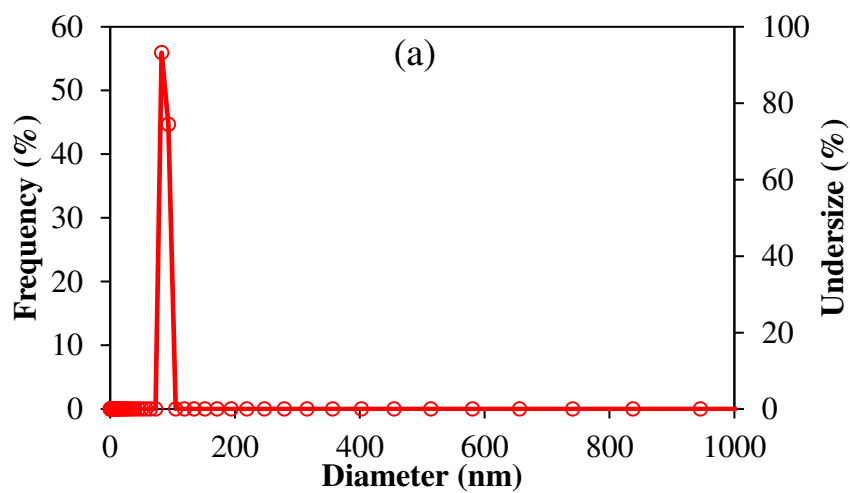


Figure 8. DLS analysis for AC (a) and AC@Fe₃O₄@SDS (b) and TEM analysis for AC@Fe₃O₄@SDS (c)

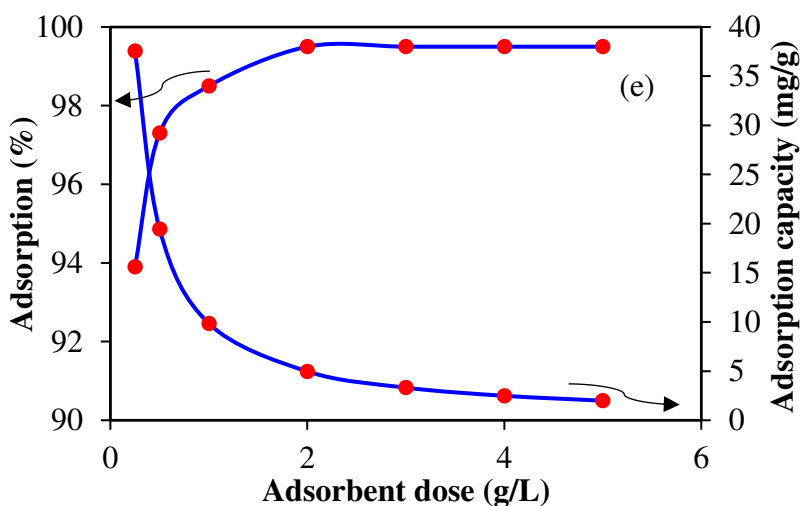
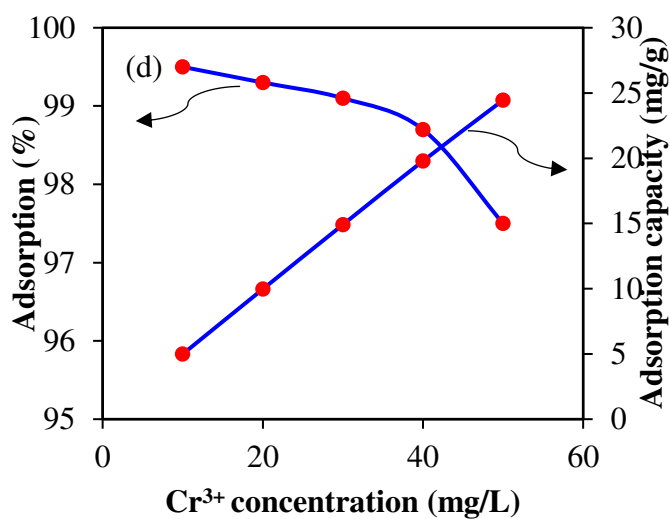
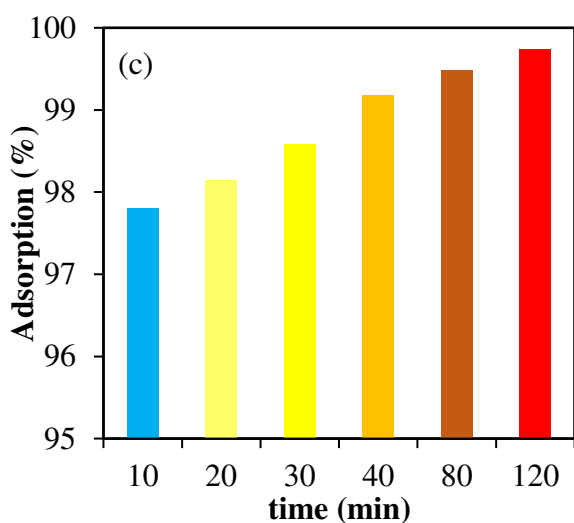
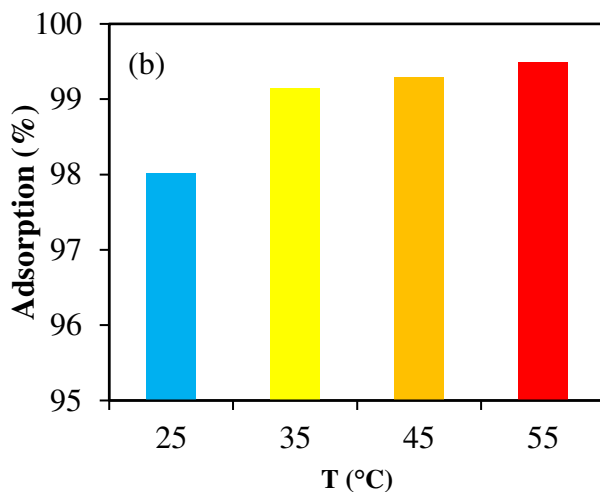
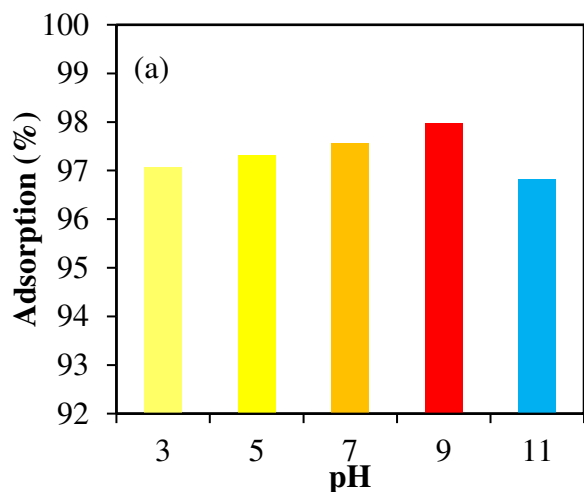


Figure 9. Impact of various parameters on uptake of Cr³⁺ using the AC@Fe₃O₄@SDS nanocomposite, including pH (a), temperature (b), time (c), chromium ion concentration (d) and nanocomposite dose (e)

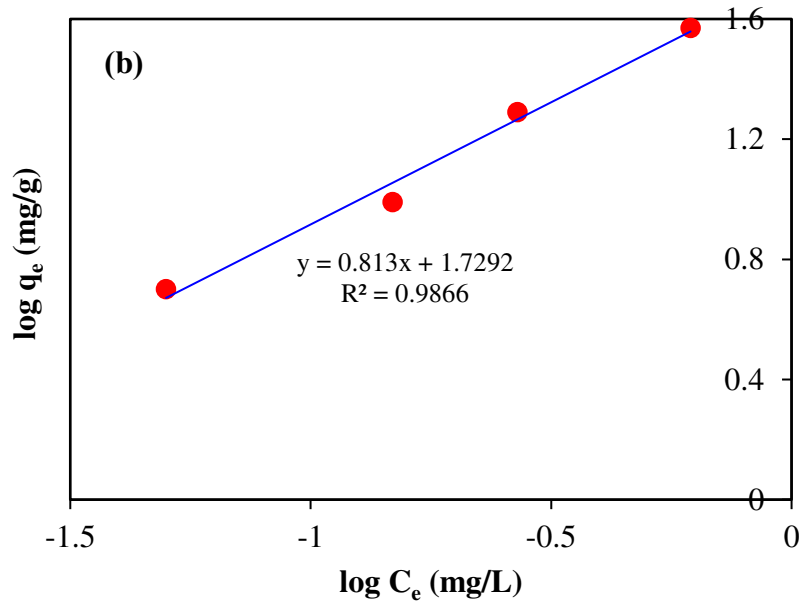
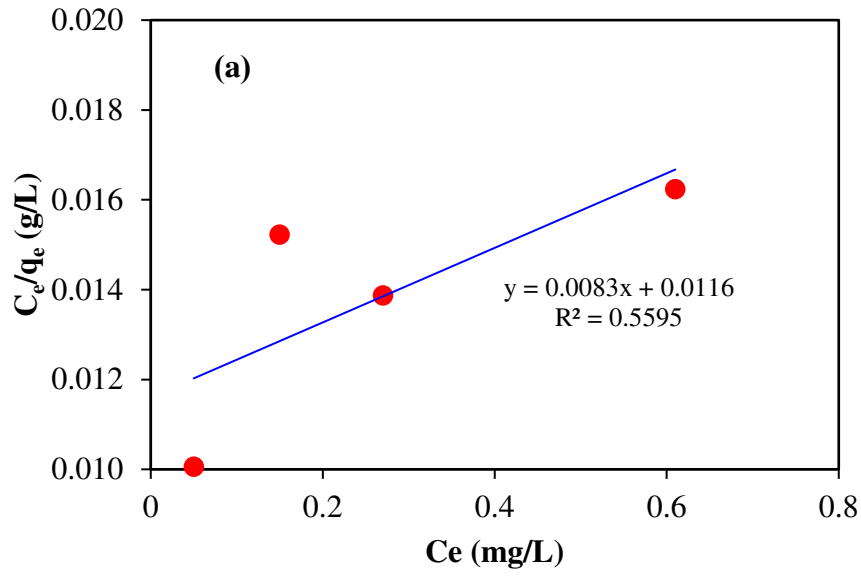


Figure 10. Sorption isotherms of Langmuir (a) and Freundlich (b) for uptake of Cr^{3+} using AC@Fe₃O₄@SDS nanocomposite

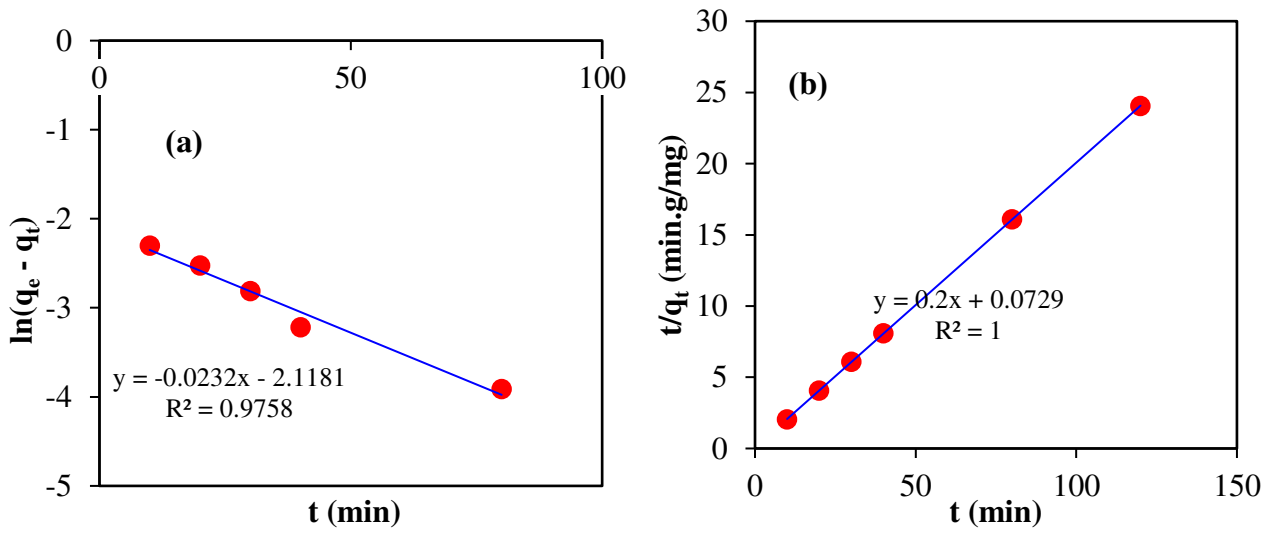


Figure 11. Sorption kinetics of QFO (a) and QSO (b) for eliminating Cr^{3+} ions using AC@Fe₃O₄@SDS nanocomposite

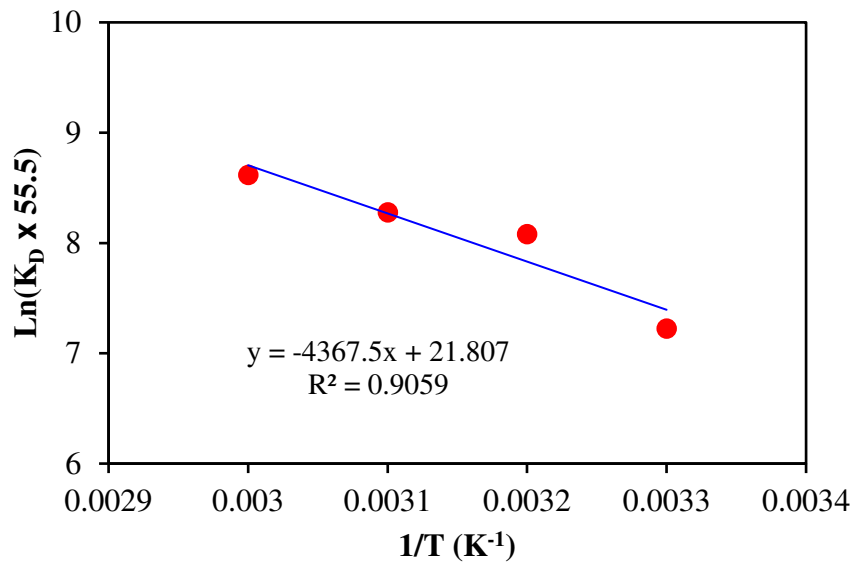


Figure 12. Thermodynamic investigation of Cr^{3+} ion sorption using AC@Fe₃O₄@SDS

603 **Table captions:**

604 Table 1. Elemental percentage in the structures of AC@Fe₃O₄@SDS before and after uptake
605 of Cr³⁺

Element	AC@Fe ₃ O ₄ @SDS		AC@Fe ₃ O ₄ @SDS+Cr ³⁺	
	Weight percent (%)	Atomic percent (%)	Weight percent (%)	Atomic percent (%)
C	13.56	22.72	18.69	33.53
O	43.27	54.44	35.05	47.19
Na	2.04	1.79	0.09	0.09
Al	–	–	0.17	0.14
Si	–	–	0.36	0.27
P	5.42	3.52	1.45	1.01
S	6.11	3.84	1.82	1.22
Cl	–	–	0.92	0.56
Cr	–	–	0.16	0.07
Fe	20.54	7.40	41.28	15.92
Ca	3.73	1.87	–	–
Mg	5.33	4.41	–	–

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609 Table 2. Constants of of Langmuir (a) and Freundlich (b) models for uptake of Cr³⁺ using
610 AC@Fe₃O₄@SDS nanocomposite

Langmuir model	Freundlich model
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R_L	$K_L(\text{L/mg})$	$q_m(\text{mg/g})$	R^2	n	$K_f(\text{mg/g})(\text{mg/L})^n$	R^2
0.123	0.71	120.48	0.559	1.23	53.6	0.986

Table 3. Comparing our results with previous work in the Cr^{3+} ion sorption

Adsorbent	$q_{\max} (\text{mg/g})$	Ref.
Bagasse fly ash	4.35	(Gupta & Ali, 2004)
Coconut shell fibers AC	39.56	(Mohan et al., 2006)
Modified peanut husk	3.02	(Li et al., 2007)
Sawdust	3.34	(Li et al., 2007)
Lignin	17.97	(Wu et al., 2008)
graphene oxide	92.65	(Yang et al., 2014)
Rind of the <i>Jatropha curcas</i>	22.11	(Gonçalves et al., 2017)
Endosperm of the <i>Jatropha curcas</i>	18.20	(Gonçalves et al., 2017)
endosperm + episperm of the <i>Jatropha curcas</i>	22.88	(Gonçalves et al., 2017)
$\text{CaO/Fe}_2\text{O}_3$	60.60	(Keshavarz et al., 2021)
$\text{AC@Fe}_3\text{O}_4\text{@SDS}$	120.48	Present study

Table 4. Kinetic parameters for uptaking Cr(III) using $\text{AC@Fe}_3\text{O}_4\text{@SDS}$

QSO			QFO		
$q_e(\text{mg/g})$	$K_2(\text{g/mg.min})$	R^2	$q_e(\text{mg/g})$	$K_1(1/\text{min})$	R^2

5	0.55	1	0.12	0.0232	0.97
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619 Table 5. The thermodynamic parameters for uptaking Cr(III) using AC@Fe₃O₄@SDS

ΔG° (kJ/mol)				ΔH° (kJ/mol)	ΔS° (J/mol)
298 K	308 K	318 K	328 K	36.31	181.30
-17.92	-20.70	-21.77	-23.52		

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