



Removal of hexavalent chromium from aqueous solution by granular and powdered Peganum Harmala



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ABSTRACT

In this paper, batch removal of hexavalent chromium from aqueous solutions by granular and powdered seeds of Peganum Harmala was investigated. The Peganum Harmala seeds were collected and after beating slowly, separating and cleaning the Harmala seeds done using a sieve. Batch adsorption studies were performed in 100 ml Erlenmeyer flasks inside an incubator container. The main process parameters considered were pH, initial Cr(VI) concentration for PPH and GPH, adsorbent dose, and contact time. Cr(VI) was measured at a wavelength of 540 nm using a UV–vis T80+ spectrophotometer. The adsorption data were fitted well by Freundlich isotherm. The result shows that the maximum removal of Cr(VI) was observed at pH 1.5 for both adsorbents. Also, by increase adsorption dose, adsorption capacity of Cr(VI) decreased but the chromium adsorption rate increased. The amount of adsorbed Cr(VI) onto both adsorbents increased with an increase in the contact time but by increases initial concentration of Cr(VI), the amount of adsorbed Cr(VI) onto both adsorbents decreased. The results indicate that the powdered Peganum Harmala can be effective adsorbent than the granular Peganum Harmala for the removal of Cr(VI) from aqueous solution.

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1. Introduction

The discharge of heavy metals into aquatic ecosystems has become a matter of concern over the last few decades [1]. Among the different heavy metals, hexavalent chromium (Cr(VI)) is a common and very toxic pollutant [2], which based to the maximum concentration limit for Cr(VI) for discharge into inland surface waters is 0.1 mg/l and in potable water is 0.05 mg/l [3]. Cr(VI) can be by variety of industries such as electroplating, leather tanning, metal finishing, nuclear power plant, textile industries, and chromate preparation discharged into the environment [4,5]. In the environment chromium occurs mainly in the oxidation states (III) and (VI) whereas Cr(III) is essential in human nutrition (especially in glucose metabolism) as well as for plants and animals at trace concentrations [3–6]. Acute exposure to Cr(VI) causes nausea, diarrhea, liver and kidney damage, dermatitis, internal hemorrhaging, and respiratory problems. Inhalation may cause acute toxicity, irritation and ulceration of the nasal septum and respiratory sensitization (asthma) and may also increase cancer risk. Also skin contact with chromium may cause allergy, dermatitis, skin necrosis and skin corrosion [7,8].

It is therefore essential to remove Cr(VI) from wastewater before disposal into the environment. In wastewater treatment, various methods applied to remove chromium include chemical precipitation, ion exchange, electrochemical precipitation, reduction, adsorption, solvent extractions, membrane separation, concentration, evaporation, reverse osmosis and biosorption [9]. The conventional process used for removal of Cr(VI) from wastewater is reduction and its precipitation as chromium(III) hydroxide. But this procedure is not completely satisfactory and has several disadvantages like generation of a large amount of secondary waste products due to various reagents used in a series of treatments such as reduction of Cr(VI), neutralization of acid solution and precipitation. The various researches have shown that the adsorption process can be a substantial method for the removal of chromium species from aqueous solutions. For these purpose, a variety of natural and synthetic materials have been tested as chromium adsorbents [10]. The various adsorbents tested in past include activated carbon and char [11,12], the modified clay [13], rice husk ash, activated alumina, coal fly ash [14] and modified corn stalk [15], etc. Therefore, there is a need for the development of low cost, easily available materials that could allow to remove and recover Cr(VI) economically [10].

Regarding the difference in adsorption capacity of various adsorbents as well as cheapness, variety and ease of access, the use of regional adsorbents is studied to evaluate their applicability. *Peganum Harmala* is a self grown and common plant that grown mainly in Throughout Iran especially the surrounding area of

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Birjand city and is easily prepared and used. Literature survey by the author in most of the peer reviewed journals indicated that adsorption study of Cr(VI) with *Peganum Harmala* seed as an adsorbent has not been investigated and this is the first such study undertaken by the authors.

Thus, the aim of this study is the use of granular and powdered seeds of *Peganum Harmala* in Cr(VI) removal from wastewater and also to compare the efficiency of granular and powdered *Peganum Harmala* seeds in the removal of Cr(VI).

2. Materials and methods

2.1. Preparation of adsorbent

In season of drying of *Peganum Harmala*, *Peganum Harmala* seeds with their sheath was collected from the local area and after beating slowly, separating and cleaning the *Harmala* seeds done using a sieve. The cleaned seeds were kept for 3 days in the sun until it completely dry.

Granular seeds with 10 mesh size were isolated by sieve and were used as the granular *Peganum Harmala* (GPH). For prepare of powder of *Peganum Harmala* seed (PPH), Granular seeds were powdered by grinder and 60 mesh size particles were isolated and were kept in a special container away from moisture and in times of need they were used as adsorbent. Then, the prepared adsorbents were characterized for their morphological structure, specific surface area, and surface functional groups.

2.2. Experimental

This study was investigated in Batch system. Batch adsorption studies were performed in 100 ml Erlenmeyer flasks inside an incubator container. The contents of all Erlenmeyer flasks were mixed thoroughly using magnetic stirrers with a fixed setting to achieve a constant speed. A stock solution of 1000 mg/l Cr(VI) was prepared by dissolving 2.8286 g $K_2Cr_2O_7$ in 1000 ml double distilled water. Required concentrations of Cr(VI) standards were prepared by appropriate dilution of the above stock Cr(VI) standard solution. For every examination, at first 50 ml of samples with determinate concentration was added into the Erlenmeyer flask. For regulating the pH range, it was used the HCl or NaOH, 1N addition of adsorbent were maintained throughout the experiments. Then having added the determinate dose of adsorbent was added into the Erlenmeyer flask and was shaking immediately in regulated speed by shaker. After desired contact time, the samples were filtered through Whatman No. 42 filter paper. The filtrates were analyzed for residual Cr(VI) concentration. The main process parameters considered were pH (1.5, 2, 2.5, 3, 4, 5, 6, 7, 8), initial Cr(VI) concentration for PPH (25, 50, 100, and 150 mg/l) and initial Cr(VI) concentration for GPH (5, 15, 25, 50, and 100 mg/l), adsorbent dose (1, 2, 4, 6, 8, 10, 15 and 20 g/l), and contact time (2, 5, 10, 15, 20, 30, 40, 50, 60, 70 and 80 min). For being sure about the results' accuracy, every sample was examined two times and the average number was reported as result.

The amount of Cr(VI) adsorbed onto the sorbent, q_e (mg g^{-1}) was calculated as follows:

$$q_e = \frac{(C_0 - C_e) \times V}{M} \quad (1)$$

where C_0 (mg/l) and C_e (mg/l) are the initial and equilibrium concentrations of Cr(VI), respectively. q_e (mg/g) is the amount of Cr(VI) adsorbed, M (g) the mass of the adsorbent and V (l) is the volume of the liquid phase.

The removal percentage ($R\%$) of chromium was calculated for each run by using Eq. (2):

$$R\% = \left[\frac{C_i - C_e}{C_i} \right] \times 100 \quad (2)$$

where C_i and C_e were the initial and final concentration of chromium in the solution, respectively.

2.3. Determination of chromium content

The pH of point of zero charge (pH_{pzc}) of the PPH and GPH was determined by the procedure detailed by [16]. The surface functional groups of PPH and GPH were determined using Fourier transform infrared spectroscopy (FTIR-KBr) at wave numbers ranging from 400 to 4000 cm^{-1} with cm^{-1} resolution. The BET specific surface area and the pore size of the PPH and GPH was determined using the N_2 adsorption/desorption method at $-196^\circ C$ [17].

A colorimetric method was used to analyze the Cr(VI) concentration of the samples. Cr(VI) was measured at a wavelength of 540 nm using a UV-vis T80+ spectrophotometer according to method presented in standard Methods for the Examination of Water and Wastewater [18]. All the primary chemicals used in this study were of analytical grade and were obtained from Merck, Germany. Statistical analyses were performed using Excel software.

3. Results and discussion

3.1. Adsorbent characteristics

The pH_{pzc} both PPH and GPH were determined from the titration curve to be approximately 6.4, indicating that a positive surface charge for a solution pH below 6.4 and a negative surface charge for a solution pH greater than 6.4. Scanning electron micrographs (SEM) of the PPH and GPH are shown in Fig. 1. It is clear from the SEM figures that the PPH surfaces were rough and contained pores of different size and considerable but the GPH particles were relatively smooth and contained deep pores. The specific BET surface area of the PPH and GPH were 7.8 and 1.15 m^2/g , respectively demonstrating that the specific surface area of the PPH has approximately 7 times greater than the GPH. Therefore, it is clear from these data that by powdering GPH, its specific surface area dramatically increase. But in general, we can say the specific surface area both of the PPH and GPH were relatively low and it shows that adsorption process is not only a physical operation.

FTIR spectra analyses at wave numbers ranging from 400 to 4000 cm^{-1} of the PPH and GPH fresh and used were undertaken and results are indicated that there are a number of important functional groups on the surface of the adsorbents. There is a wide adsorption band on fresh adsorbent for example at wave numbers between 3000 and 3633 cm^{-1} (with a maximum at 3400 cm^{-1}), which is representative of free and bonded hydroxyl groups ($-OH$) bonded to the surface of fresh adsorbents [13,17,19]. Moreover, a board adsorption band at wave numbers ranging 2800 to 3000 cm^{-1} (with a maximum at 2931 cm^{-1}) was observed in the spectra of fresh adsorbents, demonstrating that C-H groups are present on the surface of PPH and GPH [13]. Also a wide adsorption band at wave numbers ranging 1300–1500 cm^{-1} may related to the presence of phenolic groups [17]. There is a number peak at wave numbers ranging 1550–1700 cm^{-1} , indicating that the presence of carbonyl groups (C=O) on the surface of fresh adsorbents [9,20].

In Fig. 2a, by comparison of PPH fresh and used the role of functional groups is significantly visible. Comparing these two shows that fresh PPH peaks after reaction has decreased significantly that indicating the activity and role of functional groups in Cr(VI) adsorption exists in solution. Whit due attention to this issue can be said that the adsorption of Cr(VI) is a chemical adsorption

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