

# Natural organic matter adsorption from aqueous solutions using powdered activated carbon

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## Original Article

### Abstract

**Introduction:** Due to environmental and human health concerns related to humic acid (HA), mainly leading to disinfection by-products creation, removal or reduction of this matters is a necessary for drinking water suppliers. Based on resistive economy map road and move to self-sufficiency, it is necessary to produce our need in water treatment industry. One of the main consumed material is activated carbon that mainly imported to the country. In order to qualifying the produced activated carbon in HA adsorption, the present study was conducted to determine the capacity of powdered activated carbon (PAC).

**Methods:** A type of PAC was provided, then to finding the effective parameters in adsorption process, the tests of BET were done on samples; to determine special surface area (SSA), total pore volume (TVP) and mean pore diameter (MDP) of PAC pores. In a batch system, the experimental parameters were studied on 48 samples using the PAC which include; contact time (5-120 min), initial HA concentration (0.5-10 mg/L), initial adsorbent dose (0.5-20 g/L) and pH (3-11). Then the most conventional models for adsorption isotherm (Freundlich and Langmuir) and kinetic (pseudo first and -second-order) were evaluated.

**Results:** Based on BET test, the SSA was 161.53 m<sup>2</sup>/g, TVP and MDP were 0.2016 cm<sup>3</sup>/g and 4.9919 nm respectively. The maximum removal rate for HA was 97.65%, and the optimum conditions were as; equilibrium time of 10 min, initial HA concentration of 5 mg/L, adsorbent dose of 10 g/L and pH 7. The fittest adsorption isotherm and kinetic model were Freundlich isotherm (R<sup>2</sup>=0.963) and pseudo-second-order kinetic model (R<sup>2</sup>=0.999) respectively.

**Conclusion:** The results showed that produced PAC is able to remove HA as much as 97.65% in a reasonable time and dose range. Thus we can relying on production of this carbon, so it was no need of import of similar foreign products.

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## Introduction:

Organic matters are considered as the portion of harmful compounds that present in surface water and groundwater. These compounds may have be a natural or synthetic source (1,2). Natural organic matter (NOM) is a combination of anionic molecules with carboxylic and phenolic surface functional groups. In other words, NOM is a mixture of organic materials including humic acid (HA), fulvic acid, hydrophilic acid, protein, lipid, hydrocarbons and amino acids. Humic substances (mainly humic and fulvic acids) is the main compound of NOM (3,4). The presence of NOM in drinking water can be lead to problems such as color, odor and formation of disinfection by-products (2). The most important of disinfection by-products can be noted to the trihalomethanes (THM) and haloaceticacids (HAA), which are suspected carcinogen (5). These carcinogenic compounds have potential of mutagenic and cause problems such as cancers of the bladder, kidney, colon and small intestine in humans (6). In addition, NOM can be connected to the synthetic organic contaminants and heavy metals and so pass through treatment units and distribution network (4).

Therefore, the removal of natural organic compounds from aquatic environments has been one of the concerns water supply specialists and investigated in several studies.

There are many methods for removal of NOM from aqueous solutions that include coagulation-flocculation, aeration, oxidation, ion exchange, adsorption and membrane filtration (5). Most of these methods have high costs and operation and maintenance problems. Humic substances have large molecules, thus leading to the closing of the membrane pores in the membrane methods. Adsorption among processes employed in water treatment, has the most advantages including simple operation, while no need to add chemicals to water and lack of sludge production (1,7). Many absorbent has been applied for removal of the HA including nano supported oyster shell (8), ceramic membranes (9), magnetic nano particles (10), TiO<sub>2</sub>/activated carbon composites (11) and Surfactant modified zeolite (12).

The objective of this study was to determine the efficiency of PAC to adsorption of NOMs as well as HA adsorption capacity of PAC. The effect of

several important parameters such as contact time, initial HA concentration, adsorbent dose and pH was also discussed. In addition, the modeling and adsorption kinetics and isotherms were determined.

## Methods:

This experimental cross sectional study was conducted in bench scale and was contained the following steps; the study was done on 48 samples taken from synthetic water which prepared from a stock solution.

Chemicals: HA was obtained from Acros (USA) Company. Other chemicals used in this study (sodium hydroxide, perchloric acid, potassium hydroxide and potassium chloride) were purchase from Merck company. PAC was also made in Iran. The specific surface area (SSA), total pore volume (TVP) and mean pore diameter (MDP) of the PAC was calculated by BET method.

Preparation of stock solution: To prepare a HA stock solution of 1000 ppm (according to the purity of purchased HA powder), was weighed 0.5 g of HA, then it was purified by the following procedure: a) HA was purified using KOH 0.2 M and KCl 0.3 M and the obtained solution was stirred for 4 to 5 h, then centrifuged to remove insoluble materials such as humin. The supernatant was acidified using the HCL to receiving pH=1.5 to HA coagulate, and the supernatant centrifuged again. b) Relatively purified HA, then a solution of bi-distilled water and HCL (pH=3) and shaken and centrifuged. c) Then, add bi-distilled water to deposits, and was stirred for 3 hours again. Finally, the HA was transferred to a balloon 250 ml and then the resulting solution was kept in the dark and at 4°C (13).

Determination of equilibrium time: To determine the equilibrium time, 10 gr of adsorbent was added to 100 ml of HA solution with fixed HA concentration of 3mg/l, then the solution was agitated (220 rpm) for 5, 10, 20, 40, 80, 100, 120 minutes. In order to the particle deposition, the samples were centrifuged for 10 min at 3500 rpm. Then, the sampels were passed from filter paper with 0.45 micron pore size filter to PAC separation of HA solution. Finally, the remaining HA

concentration in the filtered solution was determined by spectrophotometer at 254 nm (model of SP-1900 UV) (14).

**Determination of the HA optimum concentration:** After determination of the equilibrium time, effect of initial HA concentration was studied in range of 0.5-10 mg/l from HA, pH value of 7, fixed adsorbent dose of 10g/l and equilibrium time. Finally, HA concentration of the solution was measured as above.

**Determination of the adsorbent optimum dose:** The effect of adsorbent dose on the HA removal was studied using an optimum HA concentration of 5mg/L, pH value of 7 and equilibrium time, while the dose of activated carbons varied from 0.5 to 20g/L. Finally, after shaking the solution was centrifuged and HA concentration of the supernatant was determined as described above.

**Determination of the adsorption optimum pH:** The effect of pH on HA removal was studied in the pH range of 3 to 11, HA optimum concentration, adsorbent optimum dose and equilibrium time. Input pH, was adjusted by adding HCL 0.1 M and NaOH 0.1 M to the experimental solutions. Then, HA concentration of the supernatant was determined.

## Results:

**Characterization of the adsorbents:** The SSA, TVP and MDP of the PAC was observed using BET methods (Figs.1 (a)) Table 1 summarizes characterization of PAC using the BET nitrogen adsorption analysis. As it clear from the BET experimental results, SSA was 161.53 m<sup>2</sup>/g, TVP was 0.2016 cm<sup>3</sup>/g and MDP was 4.9919 nm.

Adsorption and desorption isotherm Chart (Fig.1(b)). Also showed that at low pressures, adsorption occurs which is indicative the high interaction of adsorbent and adsorbate.

Figure 2. HA adsorption as a function of (a) contact time (pH: 7; initial HA concentration: 3 mg/L and adsorbent dose: 10g/L) (b) initial HA concentration (pH: 7; adsorbent dose: 10g/L and equilibrium time) (c) adsorbent dose (pH: 7; HA concentration: 5mg/L and equilibrium time) (d) pH (HA concentration: 5 mg/L; adsorbent dose: 10g/L and equilibrium time).

**Effect of contact time on the HA adsorption:** Fig. 2(a) displays the HA removal efficiency and its adsorption capacity as a function of the contact time. It can be seen that the adsorption of HA to PAC was rapid at the beginning (0-10 min) and then decreases gradually until equilibrium. Since, there was no significant change in the HA removal efficiency and its adsorption capacity after time 10 min, therefore it was considered contact time of 10 min as equilibrium time.

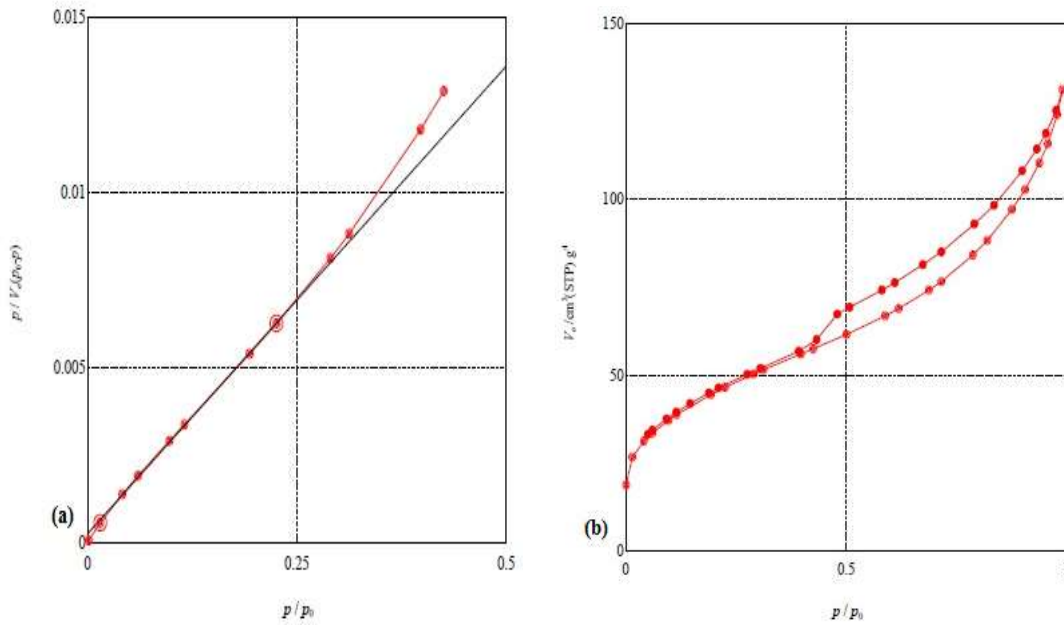


Figure 1. (a) BET plot and (b) nitrogen adsorption / desorption isotherm

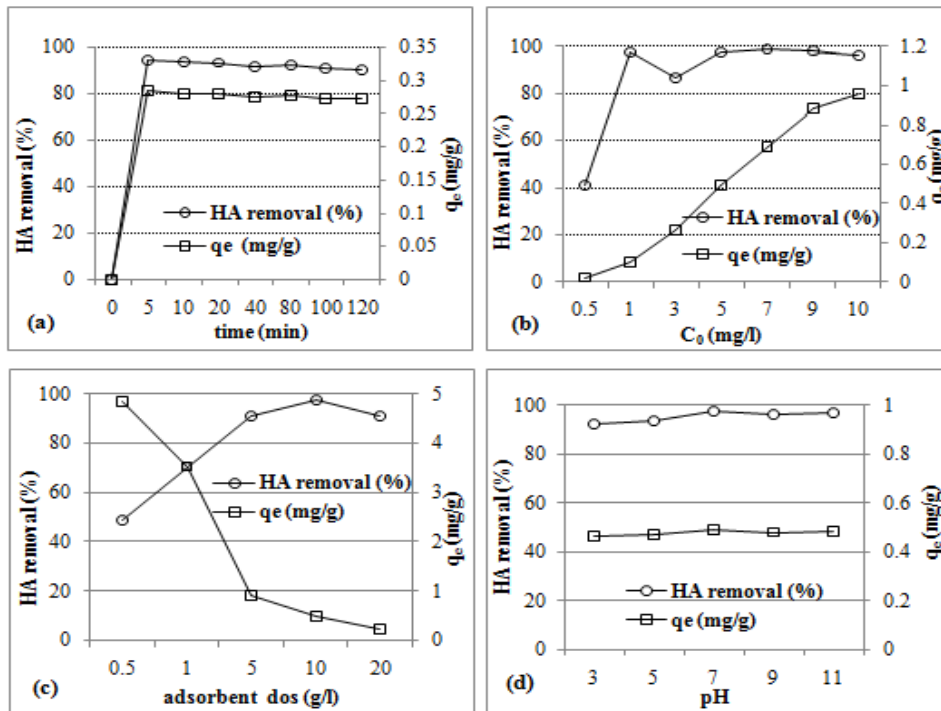


Figure 2. HA adsorption as a function of (a) contact time (pH: 7; initial HA concentration: 3 mg/L and adsorbent dose: 10 g/L) (b) initial HA concentration (pH: 7; adsorbent dose: 10 g/L and equilibrium time) (c) adsorbent dose (pH: 7; HA concentration: 5 mg/L and equilibrium time) (d) pH (HA concentration: 5 mg/L; adsorbent dose: 10 g/L and equilibrium time)

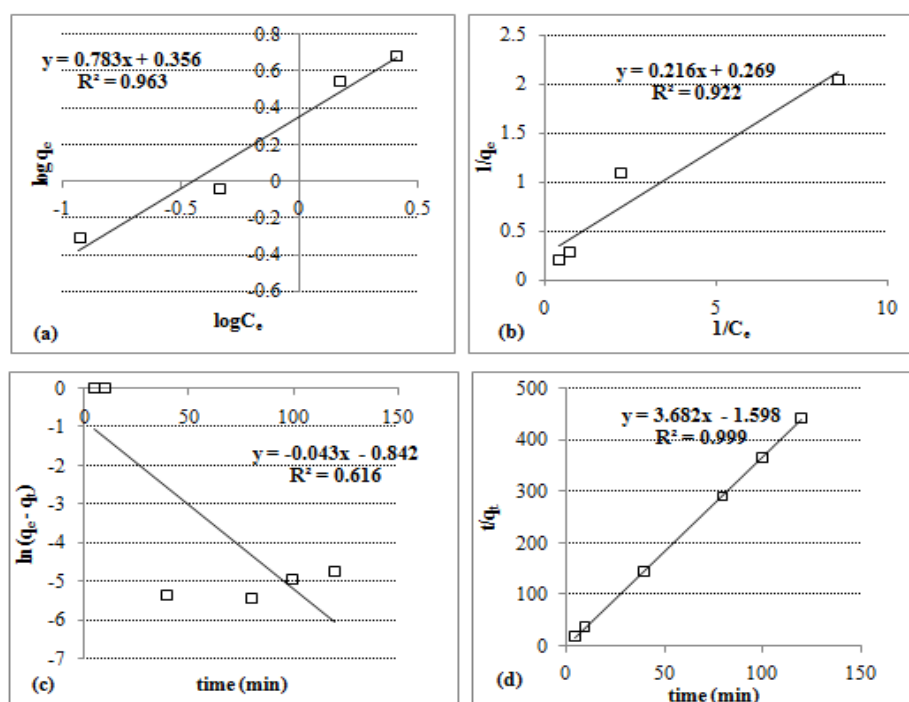


Figure 3. (a) Freundlich and (b) Langmuir adsorption isotherm (c) Pseudo-first-order model and (d) Pseudo-second-order model kinetics of HA adsorption by PAC

Table 1. Conditions and results of BET experiment

Conditions	Adsorbate	adsorption temperature (K)	Saturated vapor pressure (KPa)	Specific Surface area (m <sup>2</sup> g <sup>-1</sup> )	mean pore diameter (nm)	Total pore volume (p/p <sub>0</sub> =0.990) (cm <sup>3</sup> g <sup>-1</sup> )
Results	N <sub>2</sub>	77	88.569	161.33	4.9919	0.2016

Table 2. Adsorption isotherm parameters for HA adsorption onto powdered activated carbon

q <sub>m</sub> (mg/g)	Langmuir			Freundlich		
	b (l/mg)	R <sup>2</sup>	R <sub>L</sub>	1/n	K <sub>r</sub> , (l/mg)	R <sup>2</sup>
3.717	1.245	0.922	0.074 ~ 0.616	0.783	2.269	0.963

Table 3. Adsorption kinetics parameters for HA adsorption onto powdered activated carbon

Pseudo-first-order model			Pseudo-second-order model		
q <sub>e</sub> (mg/g)	K <sub>1</sub> (l/min)	R <sup>2</sup>	q <sub>e</sub> (mg/g)	K <sub>2</sub> (l/min)	R <sup>2</sup>
0.430	0.043	0.616	0.271	-8.50	0.999

Effect of initial HA concentration: The effect of initial HA concentration on the HA removal is shown in Fig.2 (b). It was observed that by increasing HA concentration from 0.5 to 10mg/L, HA adsorption per unit weight of adsorbent increased from 0.020 to 0.960mg/g. Similarly, the removal efficiency of HA increased from 41.16% to 96.03% by increasing HA concentration from 0.5 to 10mg/L. However, because there was no significant change in the HA removal efficiency

when the HA concentration reached higher than 5 mg/l, concentration of 5mg/l was considered as the optimum concentration of HA.

Effect of adsorbent dose: Fig.2 (c) describes the efficiency and adsorption capacity of HA to different adsorbent doses. It was observed that by an increasing in adsorbent dose from 0.5 to 20g/L, the HA removal efficiency increases from 48.55% to 91.25%, but the adsorption capacity decreases

from 4.855 mg/g to 0.228 mg/g. However, because there was no significant change in the HA removal efficiency by increasing adsorbent dose from 5 to 20g/L, adsorbent economic dose was modified from 20g/l to 10g/l.

Effect of solution pH: Fig.2 (d) shows the effect of different pH on HA adsorption by activated carbon. There was no significant change in the HA removal efficiency with increasing in solution pH, however, the maximum percentage removal of HA and adsorption capacity was achieved at pH of 7. Therefore, pH of 7 was selected as the optimum pH.

Isotherms modeling of sorption: To determination the adsorption capacity of PAC for the HA removal from aqueous solutions, Freundlich and Langmuir isotherms were studied in this study.

The linear form of Freundlich isotherm can be written as (15):

$$\text{Log}(q_e) = \text{Log}(K_f) + \frac{1}{n} \text{Log}C_e \quad (1)$$

Where  $q_e$  is the mass of HA adsorbed per unit weight of the adsorbent (mg/g),

$C_e$  is the equilibrium concentration HA in solution (mg/L) after adsorption.

The coefficients  $k_f$  and  $n$  represent the capacity and the intensity of adsorption respectively.

The Langmuir adsorption isotherm model is defined as (16):

$$q_e = \frac{q_m b C_e}{1 + b C_e} \quad (2)$$

And can be rewritten as:

$$\frac{C_e}{q_e} = \frac{1}{b q_{max}} + \frac{1}{q_{max}} C_e \quad (3)$$

Where  $q_e$  is the mass of HA per unit mass of adsorbent (mg/g),

$q_{max}$  is the monolayer adsorption capacity,

$b$  is the Langmuir constant related to the free energy of adsorption equilibrium concentration of HA in solution (mg/L) after adsorption.

factor  $R_L$  can be written as (4):

$$R_L = \frac{1}{1 + C_0 b} \quad (4)$$

Where  $b$  and  $C_0$  represent the Langmuir constant and initial HA concentration. Values of

$R_L$  between 0 and 1 indicate desirable conditions of adsorption.

$R_L$  values for the adsorption process were obtained from 0.074 to 0.616 for HA concentration in the range of 0.5 to 10. Therefore, these values showed that the conditions of adsorption was desirable.

Adsorption Freundlich and Langmuir isotherms parameters of HA onto PAC are shown in Table 2.

As shown in Fig. 3 (a and b); Freundlich model ( $R^2 = 0.963$ ) was better fitted than Langmuir model ( $R^2 = 0.922$ ).

Kinetics modeling of adsorption: The adsorption kinetics is important in the treatment of aqueous solution, therefore was used the pseudo- first and pseudo-second- order kinetics in this study. This kinetics can be written as follows (4):

Pseudo-first- order model,

$$\text{Ln}(q_e - q_t) = \text{Ln}q_e - K_1 t \quad (5)$$

Pseudo-second- order model,

$$\frac{t}{q_t} = \frac{1}{q_e^2 K_2} + \frac{1}{q_e} t \quad (6)$$

Where  $q_e$  is the mass of solute sorbed at equilibrium (mg/g),

$q_t$  the mass of adsorbed solute at time  $t$  (mg/g),

$K_1$  the first-order equilibrium rate constant (1/min),

$K_2$  is the second-order equilibrium rate constant (1/min),

$t$  is the time (min).

Adsorption pseudo-first and pseudo-second-order kinetics parameters of HA onto PAC are shown in Table 3.

As shown in Fig.3 (c); HA adsorption by PAC followed the pseudo-second kinetic model ( $R^2 = 0.999$ ).

## Conclusion:

Effect of contact time: According to Fig.2 (a); the adsorption rate of HA was rapid at the beginning and then decreased until equilibrium. The initial removal percentage of HA was high due to the high surface area of adsorbent. Adsorption decrease in later stages can be attributed to the filling of adsorption sites or problem access of HA molecules to activated sites on the adsorbent surface



(17,18). This study supported by Mansouri et al. and Lin et al studies (3,20).

**Effect of initial HA concentration:** As it shown in Fig.2 (b); adsorption rate increases with increasing initial concentration of HA. This may be due to the increase in the mass transfer force that allows more HA molecules to be absorbed onto the adsorbent surface (19). Zhan et al. found similar results in the adsorption of HA by cetylpyridinium bromide modified zeolite (20). In contrary, Mansoury et al. found that the removal efficiency of HA by activated carbon decreased by increasing initial HA concentration from 2 to 30 (3).

**Effect of adsorbent dose:** Based on Fig.2 (c); by an increase in adsorbent dose the HA removal efficiency increases, where as the adsorption capacity decreases. The increase in adsorption efficiency can be attributed to the additional number of adsorption sites, whereas the reduction in the adsorption capacity may be due to unsaturated active sites of adsorbent during the adsorption process (21). Mahvi et al. reported that efficiency of NOM removal increased with increase in the amount of sodium dodecyl sulfate modified-zeolite as a promising adsorbent from 0.2 to 1.2 g/l (4).

In another study, Doulia et al. evaluated the efficiency of acid-activated Greek bentonite in removal of HA and determined the influence of various parameters on HA sorption. They found that by an increase in bentonite dose from 0.4 to 12 g/L the removal rate of HA increases from 16.7% to 46%, but the adsorption capacity decreases from 8.35 to 0.766 mg/g (19).

**Effect of solution pH:** As it clear from the results, the maximum removal rate was obtained in pH of 7; as in acidic pH, ions of  $H_3O^+$  compete with the dissolved ions of HA to attach on the adsorption sites, so adsorption sites will filled, which leads to reduction in the adsorption capacity. In addition, the number of hydrogen ions is increased in low pH that competes with positive ions of organic compounds and reduce the adsorption of organic compounds. On the other hand, due to the repulsive force between the anions present in organic compounds and  $OH^-$  charges in surface of adsorbent adsorption will reduced at pH

higher than 7. Therefore we have the highest removal rate in the range of pH 7. Tilaki et al. examined dissolved organic carbon removal with natural and cationic surfactant modified and showed the highest removal was obtained at pH of 7 (1).

The results showed that PAC can be used as an effective absorbent for HA removal from aqueous solutions. Based on BET test, the SSA was 161.53  $m^2/g$ , TVP and MDP were 0.2016  $cm^3/g$  and 4.9919 nm respectively. The maximum removal rate for HA was 97.65%, and the optimum conditions were as; equilibrium time of 10 min, initial HA concentration of 5 mg/L, adsorbent dose of 10g/L and pH 7. The fittest adsorption isotherm and kinetic model were Freundlich isotherm and pseudo-second-order kinetic model respectively. Thus we can relying on domestic production of this carbon, so it was no need of import of similar foreign products.

**Limitations:** Due to the inadequacy of financial resources, did not exist the possibility of comparing these types of activated carbon with all kinds of imported activated carbon available on the Iran market.

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## جذب مواد آلی طبیعی از محلول های آبی با استفاده از کربن فعال پودری

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مجله پزشکی هرمزگان سال بیستم شماره چهارم ۹۵ صفحات ۲۴۹-۲۴۱

### چکیده

**مقدمه:** به دلیل نگرانی های زیست محیطی و بهداشتی بشر مربوط به اسید هیومیک که عمدتاً می توان به تولید محصولات جانبی گندزایی نسبت داد، حذف یا کاهش این مواد از منابع آب آشامیدنی الزامی است. با توجه به خط مشی اقتصاد مقاومتی و حرکت به سوی خودکفایی، تولید مواد مورد نیاز در صنعت تصفیه آب الزامی است. از جمله مواد مهم مصرفی، کربن فعال می باشد که عمدتاً به کشور وارد می شود. از آنجایی که کربن فعال در جذب اسید هیومیک مؤثر است، لذا این مطالعه با هدف تعیین ظرفیت کربن فعال پودری انجام شده است.

**روش کار:** به منظور تعیین پارامترهای مؤثر در فرآیند جذب سطحی مانند مساحت سطح ویژه، حجم کل منافذ و میانگین قطر منافذ کربن فعال پودری، آزمایش BET انجام گرفت. پارامترهای آزمایشگاهی مانند زمان تماس (۵-۱۲۰ min)، غلظت اولیه اسید هیومیک (۰/۵-۱۰ mg/L)، دوز جانب (۰/۵-۲۰ g/L) و pH (۳-۱۱) توسط کربن فعال پودری داخلی به صورت ناپیوسته بر روی ۴۸ نمونه مورد مطالعه قرار گرفت. سپس متداول ترین مدل های ایزوترم (فروندلیچ و لانگمویر) و سینتیک جذب (شبه درجه اول و شبه درجه دوم) مورد بررسی قرار گرفتند.

**نتایج:** نتایج آنالیز BET بیانگر آن بود که کربن فعال پودری نوع ایرانی دارای مساحت سطح ویژه  $161/53 \text{ m}^2/\text{g}$ ، حجم کل منافذ  $0/2016 \text{ cm}^3/\text{g}$  و میانگین قطر منافذ  $4/9919 \text{ nm}$  می باشد. بیشترین درصد حذف اسید هیومیک،  $97/65$  درصد بود و شرایط بهینه نیز در زمان تعادل تماس  $10 \text{ min}$ ، غلظت اولیه اسید هیومیک  $5 \text{ mg/L}$ ، دوز جانب  $10 \text{ g/L}$  و pH خنثی بدست آمد. بررسی مدل های ایزوترم و سینتیک جذب نشان داد که حذف اسید هیومیک توسط کربن فعال به ترتیب از مدل فروندلیچ ( $R^2 = 0/963$ ) و مدل شبه درجه دوم ( $R^2 = 0/999$ ) پیروی می کند.

**نتیجه گیری:** نتایج نشان داد که کربن فعال پودری قادر به حذف اسید هیومیک به مقدار  $97/65$  درصد در یک زمان و دوز جانب منطقی می باشد. بنابراین ما می توانیم بدون نیاز به واردات محصولات خارجی مشابه، نسبت به تولید این نوع کربن خودکفا شویم.

**کلیدواژه ها:** مواد آلی طبیعی، کربن فعال پودری، جذب سطحی، محیط آبی

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