Adsorption properties of activated carbon fiber for highly effective removal of methyl orange dye

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Abstract. A commercial activated carbon fiber (ACF) was applied as efficient adsorbents for adsorption of methyl orange (MO) dye in aqueous solution. The surface morphology and surface area were characterized by scanning electron microscopy (SEM) and \(N_2\) adsorption, respectively. The results showed that ACF had good adsorption capacity on MO dye due to the large surface area and excellent pore size structure. In addition, the adsorption property of MO dye in aqueous solution onto ACF was investigated as a function of the initial dye concentration, pH value and temperature of solution. It’s observed that the adsorption capacity of ACF for MO dye increased with the increases in the initial dye concentration and the temperature of solution, but the pH value had no significant effect on ACF adsorption performance. Langmuir, Freundlich and Temkin isotherm models were employed to fit the equilibrium isotherm data, and the Langmuir model fitted the experimental data best implying the homogeneity of the adsorbent surface sites. Besides, thermodynamics parameters, Gibbs free energy \(\Delta G^0\), entropy \(\Delta S^0\) and enthalpy \(\Delta H^0\), were calculated, and the values indicated that the physisorption process of MO dye onto ACF was spontaneous, endothermic and attractive.

1. Introduction

The environmental pollution problems of heavy metal ions, chemical oxygen demand (COD), ammonia nitrogen, petroleum, dyes in water have caused widespread concerns [1-3]. Among them dye-containing effluents discharged from industries such as feedstuffs, textile, leather, paper, cosmetics, etc, seriously contribute to coloring and COD [2, 4]. Moreover, most of the dyes can cause allergy, dermatitis and skin irritation and can also provoke cell mutation [5]. Azo dyes belong to a class of organic compound with a complex aromatic molecular structure that can provide firm and bright color to industrialized products. Due to their complex structures, they are toxic, stable and non-biodegradable [6-9]. Therefore, it’s urgent to search for efficient and economical treatment of dye-containing wastewaters. Adsorption disposal is justified as one of the most effective technologies for effluents remediation by the characteristics of convenience, high sorption efficiency, low cost and wide adaptability [5, 10-11].

Activated carbon fiber (ACF), as an important classification of adsorbents, has drawn the worldwide attention in recent years because of the higher adsorption rate from the liquid or gas phase [12-14]. And due to its relatively larger specific surface area, more uniform micropore size distribution, much higher
sorption capacity than other adsorbents and excellent recycling performance [2-3, 15], ACF is attractive alternative adsorbent to remove organic contaminants including dye pollutants in aqueous effluents [14, 16-19].

In this work, activated carbon fiber (ACF) was employed to investigate the adsorption/removal of methyl orange (MO) dye from an aqueous solution. The influence of various operating parameters, including the initial dye concentration, pH value of solution and temperature, is studied and optimized. In order to better understanding the mechanism of adsorption, spontaneity, and heat of adsorption of methyl orange, thermodynamic calculations of the adsorption process were studied. Moreover, adsorption isotherms were evaluated and reported as well.

2. Experimental

2.1. Materials
All the chemical reagents used in the work were of analytical grade. All solutions were prepared with deionized water. The commercial viscose-based activated carbon fiber (ACF) was purchased from Jiangsu Sutong Carbon Fiber Co., Ltd. (China), which was cut into $15 \times 15$ mm pieces, rinsed with deionized water while stirring, then dried overnight at 373 K.

2.2. Characterization
Nitrogen adsorption isotherms were obtained by BET surface area apparatus (Micromeritics, model TriStar II 3020) at 77 K. The specific surface areas and pore size distribution calculated from the nitrogen adsorption isotherms by BET and BJH method. The surface morphologies of ACF were analyzed by scanning electron microscopy (SEM) (LEO, model 1530VP) working under accelerating voltage, 0.1-30 kV.

2.3. Batch equilibrium studies

2.3.1. The effect of the initial dye concentration. The effect and optimum of the initial dye concentration on the ACF adsorption performance was investigated by adding the same dosage of ACF to various initial concentrations of methyl orange (100-600 mg/L) at the contact time of 2 h and pH=6.

2.3.2. The effect of initial pH. To determine the effect of the initial dye solution pH on the ACF adsorption process, the pH value range (2-10) of the MO solutions was set and the experiments were performed on fixed ACF dosage of 100 mg/L, the initial dye concentration of 100 mg/L, and the contact time of 2 h.

2.3.3. The effect of temperature. The effect of the temperature on the ACF’s equilibrium adsorption capacity of MO dye was studied at different solution temperatures (298, 308 and 318 K), the initial dye concentration of 300 mg/L, contact time of 2 h, ACF dosage of 100 mg/L and pH=6. Then, from the experimental solutions samples were obtanied and the efficiency were assessed.

In the operating process of the experiments, the liquid methyl orange solution was seperated from the ACF solid phase at the end of the adsorption equilibrium period for later analysis of the MO dye concentration determined using a spectrophotometer at $\lambda=464$ nm. The amount of MO adsorption at equilibrium $q_e$ (mg/g) was calculated with the following equation:

$$q_e = \frac{(c_0 - c_e)}{w}$$

(1)

And the removal efficiency $\eta$ of each methyl orange dye was calculated as follows:

$$\eta = \frac{(c_0 - c_e)}{c_0} \times 100\%$$

(2)
Where V (L) is the volume of the dye solution and w (g) is the mass dosage of ACF used, C₀ and Cₑ (mg/L) are the liquid phase concentrations of methyl orange at initial and equilibrium, respectively.

3. Results and discussion

3.1. Characterization

It’s reported that large surface area and pore volume can enhance pollutant diffusing onto the adsorbent surface [20-22]. Table 1 lists the parameters of the properties of ACF measured by nitrogen adsorption such as the total surface area, as well as the pore volume and pore diameter. The molecular size of the target dye pollutants MO is $1.47 \times 0.53 \times 0.53 \text{ nm}$, smaller than the average pore diameter of ACF (2.39 nm), indicating that the MO dye molecules can easily enter the inner space of ACF and then disperse, which agrees with the high adsorption of ACF in the later experimental period.

Table 1. Physical properties of ACF

| Surface area (m² g⁻¹) | Pore volume (cm³ g⁻¹) | Pore diameter (nm) |
|-----------------------|-----------------------|--------------------|
| 1278.36               | 0.809                 | 2.39               |

Scanning electron microscopy (SEM) images were used to characterize the surface morphology of ACF as shown in Fig. 1. It can be seen from Fig. 1 that the ACF washed by deionized water is tight beam and relatively smooth with few fine particles or impurities. Also it’s evident that there are many longitudinal stripes and groove structures on the surface of ACF with transverse stripes covered on the fiber surface as well.

Figure 1. SEM images of ACF.

References are cited in the text just by square brackets [1]. Two or more references at a time may be put in one set of brackets [3, 4]. The references are to be numbered in the order in which they are cited in the text and are to be listed at the end of the contribution under heading references, see our example below.

3.2. Batch equilibrium studies

3.2.1. Effect of initial dye concentration. The effect of initial dye concentration for the adsorption of MO onto ACF samples was depicted in Fig. 2. As a result, the uptake capacity of MO dye increased from 95.11 mg/g to 338.63 mg/g and the removal efficiency decreased from 95.11 % to 56.44 % with the increase of the initial dye concentration from 100 mg/L to 600 mg/L. The high adsorption capacity of the ACF adsorbent from the aqueous solution is primarily due to the great number of active sites available for adsorption which exists on the surface and inner space of the ACF.
3.2.2. Effect of initial pH. The pH of the MO dye solution affects not only the surface charge of the adsorbents, the degree of ionization of the materials and the dissociation of functional groups (-OH, -COOH, -NH₂) [23] on the active sites of the ACF surface, but also the structure of the dye molecule [5, 24-25]. To study the influence of pH value on ACF adsorption of MO dye, a series of experiments had been performed at different pH values (from 2 to 10) keeping all other parameters constant (shown in Fig. 3). It was observed from Fig. 3 that the adsorptivity kept identical both in acid and basic conditions, meaning that the initial pH exhibited little effect on the MO dye onto the ACF adsorbent, which is agreement with Ayranci’s study [26].

When the pH value of solution is acid, MO is usually present in the form of sulfonic acid, so MO is mainly absorbed by the positively charged groups such as carboxyl groups on the surface of ACF, while the pH value of solution is basic, MO is usually present in the form of sodium sulfonate, so MO is mainly absorbed by the negatively charged groups such as -NH₂ in pore channels of ACF [27]. And this result made the ACF adsorbent a promising material in dye-containing wastewater treatment.
3.2.3. Effect of temperature. It’s considered that the temperature might affect the suitability of the adsorbent if the percentage removal of a certain pollutant from an aqueous solution is temperature dependent. Various dye-containing wastewaters are produced at relatively high temperatures [5, 25]. Thereby, adsorption experiments had been performed at different temperatures of 25, 35 and 45 °C respectively to determine the influence of temperature. Fig. 4 shows that with the increase in temperature the adsorption capacity increases. As is seen, with the increase in temperature from 25 to 45 °C, the uptake capacity of MO dye increases from 174.99 mg/g to 238.04 mg/g. It’s illustrated that the adsorption process of MO dye onto ACF is endothermic.

The result of the increase in adsorption capacity of ACF may be accepted by the following interpretation. As we know that the higher temperature can enhance the mobility and penetration of dye molecules within the adsorbent porous structure. Therefore, it’s helpful to overcome the activation energy barrier and enhance the rate of intraparticle diffusion [28-29].

![Figure 4](image)

**Figure 4.** The effect of temperature on the removal of MO dye by ACF (experimental conditions: 2 h, pH 6, and MO concentration 300 mg L⁻¹).

3.2.4. Thermodynamic results. The in-depth information about the energetic changes associated with adsorption process can be provided by the thermodynamic parameters. To have a better understanding of the thermodynamics of adsorption of MO dye on ACF, the adsorption was evaluated by the adsorption coefficient $K_e$, Gibbs free energy $\Delta G^0$, entropy $\Delta S^0$ and enthalpy $\Delta H^0$ using the following equations [30]:

$$K_e = \frac{q_e}{c_e} \quad (3)$$

$$\Delta G^0 = -RT\ln K_e \quad (4)$$

$$\ln K_e = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (5)$$

Where $K_e$ (L/g) is the adsorption coefficient, $R$ (J/(mol•K)) is the gas constant, and $T$ (K) is the absolute temperature, respectively. $\Delta S^0$ (J/(K•mol)) and $\Delta H^0$ (kJ/mol) are calculated from the intercept and slope of van’t Hoff plots of $\ln K_e$ vs $T^{-1}$ (Eq. (5)). Herein, the thermodynamic parameters are obtained in Table 2.
Table 2. Thermodynamic parameters for the adsorption of MO on the surface of ACF

| T/ K | K_e/ L g⁻¹ | ΔG⁰/ kJ mol⁻¹ | ΔH⁰/ kJ mol⁻¹ | ΔS⁰/ J K⁻¹ mol⁻¹ |
|------|------------|---------------|---------------|------------------|
| 298  | 1.40       | -0.84         |               |                  |
| 308  | 2.15       | -1.97         | 38.16         | 130.86           |
| 318  | 3.84       | -3.57         |               |                  |

As can be seen that Gibbs free energy ΔG⁰ is negative, and the enthalpy ΔH⁰ is positive, respectively, which indicates that the adsorption process of MO dye onto ACF is spontaneous and endothermic. Moreover, the positive value of entropy change ΔS⁰ is indicative of the random nature of the adsorption process between the solid and solution interface, also it reflects the affinity of ACF for MO dye adsorption [5, 25, 31]. The randomness at the solid/solution interface may be attributed to the more translational entropy, which was gained by the displaced water molecules during the adsorption process, as compared to that lost by the MO dye molecules [5, 32]. In addition, The absolute value of ΔG⁰ is less than 40 kJ/mol, indicating that the adsorption process of MO dye onto ACF is a physical adsorption process [33].

3.3. Adsorption isotherms

Adsorption isotherms reveal the interaction of adsorbents with adsorbates, also it’s helpful in better understanding the equilibrium distribution at solid/solution phase [5]. Thereby, it’s considered to be critical for the optimal choice of adsorbents. In this work, the Langmuir, Freundlich and Temkin isotherm models were employed to fit the equilibrium adsorption data of MO dye by ACF.

The Langmuir model [34] assumes a homogeneous adsorption surface with adsorption sites which are identical. The form of Langmuir isotherm model can be expressed as:

\[ q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \]  

Where \( q_m \) (mg/g) is the theoretical saturation adsorption capacity, and \( K_L \) (L/mg) is a constant which is related to the free energy of adsorption.

The Freundlich model [35], as an empirical equation, is based on the assumption of a heterogeneous adsorbent surface with sites which are different in adsorption energies and are not equally available. The form of Freundlich equation can be shown in Eq. (7):

\[ q_e = K_L C_e^{1/n} \]  

Where \( K_L \) is the Freundlich affinity coefficient, and \( n \) is a constant related to the adsorption intensity.

The Temkin model [36] is applied to estimate the effects of indirect interactions between adsorbate molecules assuming the heat of adsorption decreases linearly with surface coverage. It has predictive power over a wide range of concentrations and the model can be given by Eq. (8):

\[ q_e = B \ln(K_L C_e) \]  

Where \( B=RT/b \) and \( R \) (J/(mol•K)) is the gas constant, \( T \) (K) is the absolute temperature, \( b \) (J/mol) is the Temkin constant which is in connection with the heat of adsorption, respectively; and \( K_T \) (L/g) is the Temkin isotherm constant.
Three adsorption isotherm models (Langmuir, Freundlich and Temkin) fit for adsorption of MO dye on ACF (experimental conditions: 2 h, pH 6, and 318 K).

Table 3. Three adsorption isotherm models (Langmuir, Freundlich and Temkin) parameters for MO dye on ACF

| Isotherm models | Parameters     | Value   |
|-----------------|----------------|---------|
| Langmuir        | q_m (mg g⁻¹)   | 357.14  |
|                 | K_L (L mg⁻¹)   | 0.033   |
|                 | R²              | 0.98967 |
|                 | K_F             | 58.89   |
| Freundlich      | 1/n             | 0.31    |
|                 | R²              | 0.98266 |
|                 | B               | 59.00   |
| Temkin          | K_T             | 0.82    |
|                 | R²              | 0.96395 |

The three adsorption isotherms of MO dye by ACF adsorbents in fitting with Langmuir, Freundlich and Temkin models are presented in Fig. 5, and the isotherm parameters are obtained from the slopes and intercepts of the nonlinear fittings (shown in Table 3). It is clear that the Langmuir and Freundlich isotherm models fit the adsorption experiment data better than the Temkin isotherm model by the values of higher correlation coefficients R², which indicates that the adsorption of MO dye onto ACF from aqueous solutions proceeds by a monolayer formation [5].

4. Conclusion
In this work, the activated carbon fiber (ACF) adsorbent employed with large surface area and excellent pore structure can induce effective adsorption of MO dye in aqueous solution. The influence of varying operating parameters which affect the adsorption efficiency such as the initial MO dye concentration, pH value of solution, and temperature of solution were investigated. The adsorption capacity of ACF for MO dye increased with the increases in the initial dye concentration and the temperature of solution, but it had nothing to do with pH value. Three isotherm models, Langmuir, Freundlich and Temkin equations, were utilized to fit the equilibrium isotherm data. Compared with the Temkin model, a better agreement with the adsorption experiment data is obtained using the Langmuir and Freundlich models with the higher correlation coefficients R². Further, due to the ACF absorbent, the thermodynamic
parameters, that are Gibbs free energy $\Delta G^0$, entropy $\Delta S^0$ and enthalpy $\Delta H^0$, were evaluated. The negative value of $\Delta G^0$ and the positive value of $\Delta H^0$ and $\Delta S^0$ reflected that the adsorption process was spontaneous, endothermic and attractive of ACF for MO dye effluents, respectively. Also the physical interactions between ACF and MO could be a predominant adsorption mechanism. Thereby the adsorption of MO dye onto ACF is a physisorption process. These results would be useful in view of applications of ACF adsorbent for removing methyl orange dye in wastewater.

Acknowledgments
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