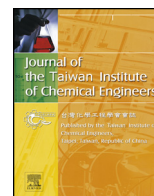




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Adsorption of a textile dye in aqueous phase using mesoporous activated carbon prepared from Iranian milk vetch

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ABSTRACT

The aim of the present study was to prepare activated carbon from milk vetch and utilize as-prepared activated carbon for sequestering Acid orange 7 (AO7) as an azo dye from batch flow mode experimental reactors. The result of SEM analysis confirmed porous structure for the activated carbon in which the major portion of the pore diameter size distribution was lower than 100 nm with frequency of 67%. As results, the adsorption of AO7 was decreased from 45.90 to 33.35 mg/g with increasing initial pH from 3 to 11, respectively. Increasing adsorbent dosage from 0.5 to 1.5 g/L resulted in increasing the decolorization efficiency (%) from 59.96 to 89.40%, respectively. The experimental data were best represented by the pseudo-second order kinetic model ($R^2 = 0.9865$) regarding intraparticle diffusion rate. Among studied isotherm models, the adsorption of AO7 onto activated carbon obeyed Langmuir model ($R^2 = 0.9907$) with a maximum adsorption capacity of about 99 mg/g. The obtained value for mean free energy (0.25 kJ/mol) indicated that the adsorption of AO7 is physical in nature. Comparatively, as-prepared activated carbon derived from milk vetch had more adsorption capacity than that of commercial activated carbon for the adsorption of AO7 at the same operational conditions.

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1. Introduction

Nowadays, water contamination due to the discharge of colored effluents containing organic dyes into aqueous environments has grown drastically [1–3]. Discharging colored effluents into such environments can cause detrimental effects on aquatic life due to their toxicity irrespective of esthetic undesirability of colored effluents [1,4–6]. There are many industries discharging organic dyes in aqueous environments such as textile, clothing, refineries, dyestuff, leather, carpet, rubber, plastic, paper and food processing [7–9]. Among different organic dyes, azo dyes are identified as carcinogenic and mutagenic compounds and can cause toxicity to aquatic organisms and human life [8,10,11]. Therefore, it is essential to remove azo dyes from colored effluents before being discharged to aqueous environments. Due to the low biodegradability of azo dyes, various physico-chemical methods such as coagulation/flocculation, chemical oxidation, chemical precipitation, electrochemical

processes, membrane technology and adsorption process have been used for treating colored effluents [8–12]. Among all above-mentioned technologies, physical adsorption onto activated carbon has proposed to be one of the most efficient and applicable approaches in terms of its simplicity, ease of operation, no generation of by-products and low sensitivity to toxic environments [6,7,10,13,14]. However, the application of commercial activated carbon has been limited because of its high cost and the need for regeneration [1,9,11,13]. Therefore, in recent years, the application of activated carbon produced from low-cost agricultural wastes has been considered as alternative adsorbents for the decolorization of colored solutions [9,11,15]. There is a growing trend on the use of carbonaceous waste materials for the production of activated carbon. Different carbonaceous waste materials such as safflower [16], hardwood [17], spent coffee [18], lignin [19], loofa sponge plant [20], pomegranate peel [10], fir wood [8], coconut husk [14] and bamboo culms [11] have been utilized as precursor for activated carbon. The application of locally available carbonaceous materials for the preparation of activated carbon can be a promising and economical approach for sequestering various target pollutants such as organic dyes in aqueous phase [13]. Thus, in the present study, the application

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of milk vetch, as a locally available plant in Iran, has been considered as precursor for activated carbon to remove Acid orange 7 (AO7) as an azo dye from aqueous solutions. Milk vetch is a large genus of about 3000 species of herbs [21], which is distributed in large quantities in Iran. Thus, it can be used as an inexpensive carbonaceous material for preparing activated carbon. Moreover, the inexpensive activated carbon can be disposed without the need for regeneration. Chemical activation is preferred over physical activation for the synthesis of activated carbon because of its simplicity, shorter activation time and better evolution of the porous structure [16]. Therefore, in the present work, chemical activation of precursor by concentrated phosphoric acid was employed. Hitherto, there has been no report on the use of milk vetch for the production of activated carbon and subsequent application for treating colored solutions. The effect of initial pH, adsorbent dosage, reaction time, adsorbate concentration and the presence of competing anions on the adsorption of AO7 was evaluated. In addition, kinetic and isotherm studies were performed to reach a better understanding of the mechanism of adsorption process.

2. Materials and methods

2.1. Chemicals

Milk vetch as precursor for activated carbon was collected from a hill near Ilam city, Iran. Commercial activated carbon, which was charcoal actiff (Molecular weight = 12.01 g/mol and solubility = 2%), was purchased from Merck, Germany. AO7 was purchased from Nasaj Sabet Co., Iran and used as received. The characteristics of AO7 are illustrated in Table 1. All other chemicals (analytical grade) were purchased from Merck, Germany. Stock dye solution was prepared (1000 mg/L) and desired concentrations of the dye were prepared through dilution of stock solution by distilled water.

2.2. Preparation of activated carbon from milk vetch

Firstly, the milk vetch shrub was washed with distilled water to remove undesirable materials and dried in sunlight for 7 days. Then, it was crushed into small pieces and dried in an oven at 100 °C until constant weight. In the following, dried milk vetch was soaked in concentrated phosphoric acid (95%) for 30 min under string for activation. The weight ratio of milk vetch to phosphoric acid was set to 1:10. The activated precursor was placed in a stainless steel vessel and heated at 600 °C for 1 h in an electric furnace. After that, activated carbon was rinsed with deionized water until the pH of rinsing water reached 6.5. The sample was placed in the oven again at 120 °C for 1 h. Finally, it was ground, sieved and particles between 60 and 120 mesh size were utilized for the adsorption of AO7 in the

subsequent experimental runs [4,6,11,13]. The sieved activated carbon was stored in a desiccator before conducting experiments.

2.3. Experimental procedure

Hundred milliliter Erlenmeyer flasks were used as batch flow mode experimental reactors to investigate the effect of studied operational parameters influencing the adsorption of AO7 onto as-prepared activated carbon. A shaker (Dragonlab, Model: SK-L330-Pro, China) was used to agitate the flasks containing AO7 for an efficient interaction between the dye molecules and adsorbent. The experimental runs were conducted according to Table 2. A control Erlenmeyer flask without activated carbon was employed to determine the adsorption of AO7 in the absence of adsorbent. All experimental runs were performed twice and the mean values were reported. Before the addition of adsorbent, the pH of colored solutions was adjusted using 0.5 M HCl and 0.5 M NaOH solutions. All experiments were carried out at room temperature (~25 °C).

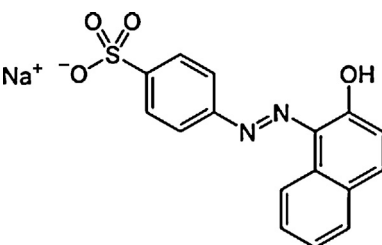
2.4. Analysis

Scanning electron microscopy (SEM) was employed to analyze structural characteristics of the as-prepared activated carbon by means of a scanning electron microscope (TESCAN, Model: MIRA3, Czech Republic). Then, one of the obtained SEM images was used to calculate pore diameter size distribution of the synthesized activated carbon using Manual Microstructure Distance Measurement software (Nahamin Pardazan Asia Co., Iran). Moreover, specific surface area of the adsorbent was determined through BET analysis (BET-Quantochrome 2000-USA). Fourier transform infrared (FT-IR) analysis was carried out using a spectrophotometer (Tensor 27, Bruker, Germany) to specify the role of surficial functional groups of the activated carbon in adsorption process. The spectra were recorded in the wavenumber between 400 and 4000/cm. Furthermore, the point of zero charge (pH_{pzc}) was measured using pH drift method to find out the surface charge of the adsorbent [5]. To measure the pH of solution, a Metrohm pH meter was used (Model: E532, Germany). At regular time intervals, the samples were collected and centrifuged for 5 min at 5000 rpm and resulted supernatants were used to determine the residual concentration of AO7 with a UV-vis spectrophotometer (DR-5000, Hach Co., USA) at maximum wavelength of 485 nm. The amount of adsorbed AO7 was calculated through the following equation:

$$q = \frac{(C_0 - C) \times V}{M} \quad (1)$$

where q (mg/g), C_0 (mg/L) and C (mg/L) are the amount of adsorbed AO7, the initial and the final concentration of AO7 in the solution, respectively. In addition, V (L) is the volume of the colored solution

Table 1
Characteristics of Acid orange 7.

C.I. name	Chemical structure	Chemical class	Molecular formula	M_w (g/mol)	λ_{max} (nm)
Acid orange 7 (AO7)		Anionic dye	$C_{16}H_{11}N_2NaO_4S$	350.32	485

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