



# Preparation of activated carbon from date sphate using microwave irradiation and investigation of its capability for removal of dye pollutant from aqueous media



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## ABSTRACT

Date sphate has been applied as a primary, abundant, cheap and available precursor to produce activated carbon for removal of a dye pollutant from aqueous solution. Phosphoric acid as an activating agent and microwave energy as a heating source have been used. Using microwave irradiation instead of furnace heating caused the decrease of operation time, saving and homogeneous heating of sample. To optimize the method of production, the effects of different parameters on the capability of the produced activated carbon for dye removal were analyzed using statistical method. The prepared activated carbon was characterized by CHN elemental analysis, SEM, N<sub>2</sub> adsorption/desorption and pH<sub>pzc</sub> methods. The ability of the prepared adsorbent for removal of methylorange and the effect of temperature and pH on removal efficiency were studied. Adsorption of methylorange was studied from kinetics and equilibrium point of view. The experimental data were fitted with different isotherms and kinetics models.

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## 1. Introduction

The extensive use of organic compounds, heavy metals, herbicides and dyes have led to the spread of hazardous substances in environment, which are penetrating into ecological cycles and threatening human life. Among these contaminants, dyes are more significant because of their complex molecular structure which generally embodies aromatic rings with various functional groups. On the other hand, they are widely used in various industries such as textile, leather, plastic, photographic, pharmaceutical and cosmetic industries and food processing [1]. These toxic materials can cause considerable damages to human health such as brain, liver, central nervous system and kidneys problems [2]. Large classes of dyes, contain azo dye compounds which are used in textile processing industries. These molecules with one or several azo (N=N) bridges linking are a serious threat for aquatic ecosystems. They

are chemically and photolytically stable which would be removed difficulty. Methylorange (Me-Org) is one of the azo dyes which is thought to be toxic if swallowed or inhaled. The structure of Me-Org is demonstrated in Fig. S1.

Additionally, water scarcity and the limitation of its supplies has caused various techniques to be applied for the removal of pollutants from aqueous solutions such as filtration, reverse osmosis, ion exchange, precipitation, coagulation, sedimentation, membrane process etc. However, the disadvantages of these methods are expensiveness, time wasting, use of chemical reagents and large volume of sludge formation. Adsorption is one of the facile techniques, which do not need special chemical solvents. Due to this advantage, adsorption has been widely used for water purification. Activated carbon with high surface area, inert nature in extreme pH conditions and presence of functional groups is an excellent adsorbent for the removal of great variety of contaminants from waste water.

Using activated carbon as catalyst [3], or more important, as catalyst supports [4] is another application of activated carbons. Despite marked advantages of activated carbons, its cost is a disadvantage. Several studies have been reported to find low-cost carbonaceous materials. Ultimately, agricultural by-products and waste materials such as rice husk [5], bamboo [6], nut [7], hazel-nut shell [8] and grape wine rhythodim [9] have been used as precursor for preparation of activated carbon.

*Abbreviations:* AC, activated carbon; Me-org, methyl orange; PAC, prepared activated carbon; DR, Dubinin–Radushkevich; SEM, scanning electron microscope; RMS, response surface methodology; DFT, density functional theory; DA, Dubinin–Astakhov; PFO, pseudo first order; PSO, pseudo second order; L, Langmuir; F, Freundlich; R-P, Redlich–Peterson.

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It is well-known that surface physical and chemical properties of activated carbon depend on, raw materials, way of manufacturing and, more significantly, heating mechanism.

Unfortunately, applying conventional heating techniques is time-consuming and increases energy and gas consumption in order to keep relatively high temperature of heated material. On the other hand, the carbon becomes damaged, due to heating and cooling cycles, and needs further treatment [10]. Another obvious problem during external heating is a thermal gradient from hot surface of adsorbent to its interior parts, which do not provide uniform temperature for different shapes and sizes of raw materials. Microwave radiation has been successfully applied for preparation and modification of activated carbons [9]. Reduction of processing time and energy consumption, uniform temperature, and rapid temperature rise, are the advantages of this technique in comparison with conventional heating methods [11].

To prepare activated carbon, two processes have been introduced: physical and chemical activation [12]. Physical activation contains two steps, first pyrolyzing raw material under inert atmosphere at a temperature above 400 °C, second activating with gases such as steam, air or carbon dioxide at temperature above 600 °C [13]. Chemical activation involves using dehydration agent such as H<sub>2</sub>SO<sub>4</sub> [8], H<sub>3</sub>PO<sub>4</sub> [9], NaOH [10], KOH [14], ZnCl<sub>2</sub> [15] and other alkaline metal compounds followed by thermal activation to create pore structures. In chemical activation, the obtained carbon yield is higher, and better developed pore structures have been seen while the temperature used in this activation is lower than that of physical activation [12].

The present study aims to introduce a new low-cost activated carbon from date sphate as an agricultural by-product. The date is produced in the southern part of Iran with export of more than 60 thousand tons per year. Date sphate is a special part of palm trees which connected date to the palm trunk (Fig. S2). Easy collecting, porous and strong structures are some of the advantages which make date sphate as powerful precursor for preparation of activated carbon.

It seems that preparation of AC from date sphate as an abundant agricultural waste leads to lower cost production. The purpose of the present work is to find out the most economical and efficient procedure for preparation of AC from date sphate with H<sub>3</sub>PO<sub>4</sub> as a chemical agent under microwave heating and to investigate its efficiency for the removal of Me-Org from aqueous solutions.

## 2. Materials and methods

### 2.1. Materials

The collected date sphate from palm groves of Boushehr city (southern part of Iran) were washed with distilled water for several times, to remove dirt particles from its surface and dried in oven at 55 °C overnight. Dried date sphates were crushed to small pieces and used as precursor and phosphoric acid (85 wt%, Merck Co.) has been applied as chemical activating agent. Me-Org (Fluka Co.), Bromothymol blue (BDH Chemical Ltd.) and Methyl violet (Merck Co.) used as adsorbate.

### 2.2. Equipment

The residual Me-Org concentration in the samples was determined by using UV/vis spectrophotometer (PG Instrument LTD Model T80). The pH measurements were carried out by a pH meter (OHAUS starter 3000). During adsorption tests a thermostat shaker (n-Bio TEK, NB-304) was utilized. Also, a microwave oven (MOLINEX, Model MVV 200130 2450 MHZ) was used for preparation of AC. The resultant activated carbon was characterized using

the scanning electron microscope (PHILIPS, X 130), while the specific surface area of the sample and pore size distribution were determined by nitrogen adsorption isotherm data (below  $P/P_0 = 0.3$  at 77.3 K, Queantachrome Nova win 2).

### 2.3. Preparation of activated carbon

The dried material (1.84 g) was soaked in phosphoric acid with different concentrations (40, 60 and 85 wt%) for 24 h at room temperature with an impregnation acid/char weight ratio of 3:1. Carbonizations of impregnated samples were carried out in microwave oven with different powers (400, 540 and 700 W) and radiation times (1–3 min) under ambient atmosphere. The carbonized samples were placed in an oven at 110 °C for 5 h. Then, the samples were washed with distilled water until the pH of washing solution reached 6–7 and put in an oven at 50 °C overnight for drying. Finally, the obtained activated carbons were finely powdered and stored in desiccator. The yield of obtained activated carbon at optimum conditions is 54%.

To study the roles of microwave power and radiation time, 5 ml of Me-Org (or methyl violet or bromothymol blue) solution with certain concentration was added to 2 mg of adsorbents which were prepared in different conditions. The samples were placed in shaker at 150 rpm and 30 °C for 16 h.

To obtain the ability of prepared carbon for removal of dye from aqueous solutions, the removal percentage was calculated by:

$$\%R = \frac{C_0 - C}{C_0} \times 100 \quad (1)$$

where  $C_0$  is the initial concentration (mg/L) and  $C$  is the residual concentration of Me-Org after certain time.

### 2.4. Characterization of activated carbon

Adsorptive properties were determined by using Me-Org as an adsorbate. Therefore, Me-Org was examined as probe molecules for achieving the adsorption capacity of adsorbent and other surface physical properties of AC were determined with N<sub>2</sub> adsorption/desorption isotherms using Quantachrome-Nova win 2 at 77.3 K. The degassing time and temperature were 7 h and 200 °C, respectively. Cumulative pore volume and average pore diameters were calculated by DFT methods. Surface area and micropore volume were measured by BET and  $t$ -plot method.

Also the morphology of the prepared activated carbon was studied by Scanning Electron Microscope (SEM).

### 2.5. Adsorption experiment

Equilibrium and kinetic tests were carried out for adsorption of Me-Org by the prepared activated carbon (PAC). In the equilibrium adsorption experiments, 5 ml of Me-Org aqueous solution with different concentrations was added to 2 mg of adsorbents. The samples were placed in shaker with speed of 150 rpm at 30.0 °C For 24 h. Then, the adsorbent was separated by centrifuge and the residual concentrations of Me-Org were determined with a UV/vis spectrophotometer at  $\lambda_{\max} = 465$  nm. The amount of Me-Org adsorbed per unit mass of adsorbent at equilibrium conditions,  $q_e$  (mg/g), was derived based on Eq. (2):

$$q_e = \frac{V(C_0 - C_e)}{W} \quad (2)$$

where  $C_0$  and  $C_e$  are initial and equilibrium concentrations, respectively (mg/L).  $V$  (L) is the volume of solution which was added to the mass of adsorbent,  $W$  (g).

Kinetic adsorption experiment was carried out by adding 2.0 mg of adsorbent to 5 ml of Me-Org aqueous solutions with certain

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