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Adsorption of methylene blue and eosin yellow using porous carbon prepared from tea waste: Adsorption equilibrium, kinetics and thermodynamics study

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ABSTRACT

Porous carbon with high surface area was synthesized from different tea precursors under thermochemical condition in presence of phosphoric acid as activating agent. The synthetic procedure was optimized under different condition of acid impregnation ratio, heating temperature, dwelling time, etc. Pore morphology, surface properties, crystalline nature and thermal stability of porous carbon (PC) were assessed by using BET, SEM, FTIR, XRD and TGA analysis. Under optimized synthetic condition, the maximum surface area of the carbon material was found to be 2054.49 $m^2 g^{-1}$ with a total pore volume of 1.747 $cm^3 g^{-1}$. Adsorption characteristics of the PC for removal of a cationic dye (methylene blue) and an anionic dye (eosin yellow) were investigated spectrophotometrically as a function of initial dye concentration and contact time, adsorbent dosage, solution pH and temperature, etc. The PC was found to be equally efficient for removal of both the dyes from aqueous solutions at neutral pH with adsorption performance of greater than 99%. The equilibrium adsorption data of both the dyes could be described well by the Langmuir isotherm equation. The maximum adsorption capacity for methylene blue and eosin yellow was found to be 402.25 mg g^{-1} and 400 mg g^{-1}, respectively. The adsorption kinetics in case of both the dyes follows a pseudo-second-order pathway. Investigations on thermodynamic parameters in the temperature range of 303–323 K revealed that the adsorption process is spontaneous and endothermic in nature.

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Introduction

Environment contamination, in general, and water pollution, in particular has become an increasingly great concern to the scientific community all around the world in recent years. The discharge of industrial wastewater containing different dye contaminants causes serious environmental problems. A variety of methods such as adsorption, coagulation and flocculation, oxidation, chemical precipitation, and so forth, have been reported for waste water treatment [1–7]. Among these, adsorption is well established and superior technique because of their low cost, the simplicity of design and availability of adsorbents. Various kind of adsorbents were used in recent years for absorptive removal of contaminants [8–40]. Activated carbon (AC) has been found to be the most widely used adsorbent for waste water treatment [25–39]. Research interests have therefore been growing for the production of porous carbon from agricultural wastes for removal of dyes from wastewater [28–39]. In the recent past, several materials from agricultural waste such as coconut coir, rice husk, wheat straw, sawdust, nutshells, etc. have been used for the preparation of AC and used as adsorbents for the removal of dyes and other pollutants from waste water systems [25–39]. Recently, we have reported our preliminary results on the synthesis of porous carbon from tea leaves and its use for chromium adsorption [40]. We report herein an improved process for the synthesis of high surface area porous carbon (PC) from tea (*Camelia sinensis*). A detail study has been undertaken to establish the most suitable condition for a high surface area porous carbon. The as synthesized PC has been utilized for adsorptive removal of both cationic dye (methylene blue) as well as anionic dye (eosin yellow). Various experimental parameters viz., initial dye concentration, contact time, adsorbent dose, solution pH and temperature were studied via batch technique. The usefulness of pseudo-first-order and pseudo-second-order kinetic models was evaluated and applicability of Langmuir and Freundlich isotherm models to understand the adsorption mechanism of both cationic and anionic dye onto

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the PC was examined. Moreover, the thermodynamic feasibility along with the spontaneity of the adsorption process in terms of the thermodynamic parameters (Gibbs energy (ΔG), enthalpy (ΔH) and entropy (ΔS)) was discussed.

Materials and methods

Preparation of adsorbent

Different form of tea (black, green and waste) used as carbon precursors were collected from commercial source and household uses. These raw materials were then washed with distilled water to remove the water soluble impurities and surface adhered particles and then dried in oven at about 60 °C to remove moisture and volatile impurities. The dried materials were then grinded to fine powder and sieved by mesh size of 60 to make fine powder of uniform size. The carbon precursor was impregnated with 85% phosphoric acid (PA) by varying the chemical ratio from 1:1 [precursor:activating agent (w/v)] to 1:3 and kept in oven at 60 °C for 3 h with occasional stirring. The acid impregnated carbon precursors were placed in a horizontal temperature programmable furnace for thermal treatment under nitrogen (N_2) atmosphere. The heating rate for all the samples were fixed on 5 °C/min. The temperature ramping was programmed in a continuous and segment wise (S) heating with a holding temperature of 1 h in each segment from 200 °C to next desired temperature with 100 °C interval. After cooling down to normal temperature, the carbonized sample was washed initially with 0.1 M HCl and then with distilled water till a neutral supernatant was obtained. A detail study was undertaken to optimize the synthetic method [41] under different condition of acid impregnation ratio, heating temperature, dwelling time, etc. All the PA treated PC samples were coded as BTPA, WBTPA and WGTPA for black tea and waste black or green tea, respectively followed by their impregnation ratio and maximum temperature with holding time in hour in case of continuous heating otherwise "S" for segment wise heating afterwards. All the studied samples were dried at 110 °C for 24 h and stored in desiccators for further analysis. Experimental results obtained for the carbon under optimized condition having maximum BET surface area are presented in this manuscript.

Adsorbates

All reagents used are of analytical grade from Merck (Germany). Stock solutions (1 g L^{-1}) of both the dyes methylene blue (MB) and eosin yellow (EY) were prepared by dissolving appropriate quantity of MB (cationic dye) of chemical formulae $C_{16}H_{18}ClN_3S$ (MW = 319.87 g mol^{-1}) and EY (anionic dye) of chemical formulae $C_{20}H_6Br_4Na_2O_5$ (MW = 691.88 g mol^{-1}) in distilled water. All test solutions of the desired concentrations were prepared by successive dilutions to get the required initial experimental concentration (100–400 mg L^{-1}). The structures of both the dyes are represented in Fig. 1.

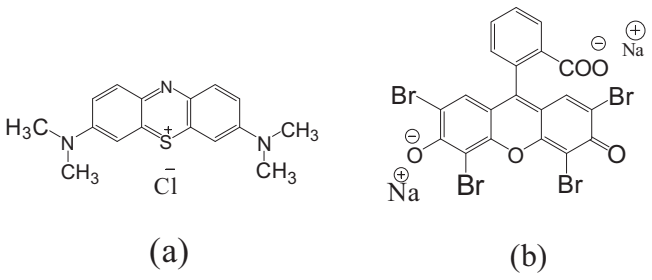

Fig. 1. Molecular structure of (a) MB and (b) EY.

Surface characterization of porous carbon

Zeta potential of the studied PC, WBTPA(1:3)400 1 h was measured using Beckman Coulter Zeta potential analyzer (Delsa™ Nano C) based on the Laser Doppler electrophoresis technique. At pH 7, the zeta potential for the studied PC was found to be −20.60 mV. The pH at which the zeta potential is zero is measured as point of zero charge (pH_{PZC}), and hence, the net charge on the adsorbent surface is zero. The pH_{PZC} of the PC, WBTPA(1:3)400 1 h was found to be 3.4. Above the pH_{PZC}, the adsorbent surface becomes negative and favored for adsorption of cations. The concentration of surface functional groups of PC was determined by Boehm titration method [42]. The amount of total basic and acidic sites as per calculation was found to be 0.529 mol/g and 0.6 mol/g, respectively.

Adsorption experiments by batch method

Adsorption equilibrium and kinetic data were obtained by batch technique individually for each dye-PC system using the concentration variation method at three different temperatures (303 K, 313 K and 323 K). Various operating parameters such as pH of the solution, adsorbent dosage, initial dye concentration and contact time and adsorption temperature on dye removal were studied. The effect of pH on adsorption of MB and EY was investigated by varying the initial pH from 3 to 11. Different adsorbent amount (1 g L^{-1}, 2 g L^{-1} and 3 g L^{-1}) were mixed in a 250 mL conical flask of dye solution over the concentration range of 100–400 mg L^{-1} and were stirred at 100 rpm in an orbital incubator shaker (laboratory Tech, LSI-1005R). At the end of predetermined time intervals, the residual dye concentration in the supernatant liquid was determined using a UV–vis spectrophotometer (PerkinElmer, Lambda 35) at its maximum wavelength (λ_{max}) of 664 nm and 517 nm for MB and EY dye, respectively. All of the experiments were performed in triplicate to check the reproducibility of data and the average of the results were used for data analysis.

The percentage removal of dyes was calculated using Eq. (1) and the concentration retained in the adsorbent phase (q_e, mg g^{-1}) at equilibrium and at time 't' were calculated using Eqs. (2) and (3), respectively.

$$\%\text{Removal} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (1)$$

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

$$q_t = \frac{(C_0 - C_t) \times V}{W} \quad (3)$$

where C_0 and C_e and C_t are the initial, equilibrium and at time t concentration (mg L^{-1}) of dye solution, V is the volume of the solution (L) and W is the mass of the adsorbent (g).

Results and discussion

Physiochemical characterization of adsorbent (PC)

BET analysis

Nitrogen adsorption desorption isotherm (Beckmann Coulter SA-3100) was used to determine the textural properties of the prepared porous carbons (Fig. 2). The nature of isotherm of all the PCs were of same type irrespective of the carbon precursor and is of type II as per IUPAC classification [43]. In case of all the isotherms, H4 type hysteresis loop was observed, which indicates the

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