



Equilibrium Isotherm and Kinetic Studies of Adsorption of Basic Green-4 on Titanium Dioxide Nanoparticles

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Abstract: Synthesis of titanium nanoparticles (TiO₂-nps) were performed by using sol-gel method. The synthesized sample was characterized by infrared spectroscopy (FT-IR), X-ray diffraction spectroscopy (XRD), atomic force microscope (AFM) and scanning electron microscope (SEM). The results indicate the presence of crystalline anatase phase TiO₂ with average particle size of 108 nm. SEM observations showed that TiO₂-nps have spherical morphology with some agglomeration. The adsorption capacity of synthesized TiO₂-nps was evaluated by using Basic green-4 dye (BG-4). The effect of different parameters, including contact time, initial concentration and temperature, on the adsorption of BG-4 dye were studied. The percent of dye removed by 0.01g of TiO₂-nps at 30°C was nearly 40%. The equilibrium data were analyzed using Langmuir and Freundlich isotherms. Freundlich isotherm fit data very well, which implies the heterogeneity of surface and that adsorption uptake of BG-4 by TiO₂-nps is multi-layers. The adsorption system obeys the pseudo-second order kinetic model and thus supports the assumption behind the model that may be due to chemisorption. The thermodynamic study showed that the nature of adsorption was exothermic and non-spontaneous.

Keywords: Titanium dioxide, Nanoparticles, Basic green-4, Adsorption isotherm.

Introduction

The wastewater discharge from textile, paper, cosmetics, plastic and rubber industries contain a variety of toxic dyes⁽¹⁾. The impact of these dyes on the environment is a major concern due to their carcinogenic and mutagenic properties^(2,3). In addition, the dyes can cause human health disorders such as allergic dermatitis and sever damage to the central nervous system^(4,5). Many available technologies have been adopted for the removal of dyes from wastewater, such as chemical precipitation, electrochemical process, ion exchange, membrane filtration, coagulation and adsorption⁽⁶⁻¹²⁾. As a less costs and high efficient method, adsorption technique has been widely used to remove dyes from industrial wastewater. Numerous of high adsorption capacity materials have been used as an adsorbents for removal of different types of colored compounds including, activated carbon prepared from different sources^(13,14), chitin and chitosan biopolymers⁽¹⁵⁻¹⁷⁾, silica^(18,19), microbial biomass^(20,21) and agricultural wastes^(22,23). Recently, nanomaterials have been explored as an adsorbents for removal of dyes and other types of organic materials from wastewater. Nanomaterials have unique size-dependant properties which relate to the high specific surface area such as, high activity associated with available active sites, short intraparticle diffusion distance and strong sorption capacity⁽²⁴⁾. Literature reports a variety of works on wastewater treatment and removal of chemical and biological compounds by adsorption with nanomaterials⁽²⁵⁻²⁸⁾. One of the most important technical nano-adsorbent is carbon nanotubes which is shown higher adsorption capacity than activated carbon on adsorption of many kinds of organic

chemicals⁽²⁹⁻³¹⁾. Metal oxide nanoparticles such as iron oxide, aluminum oxide and cerium oxide have been explored as an effective and low cost adsorbents for treatment of wastewater contaminated with heavy metals⁽³²⁻³⁴⁾. Due to its high external specific surface area and band gap (3.2 eV), nano titanium dioxide is one of metal oxides that has a wide range of applications in photocatalytic, electronic and adsorption fields⁽³⁵⁻³⁸⁾. The titanium dioxide nanoparticles as an adsorbent surface is eco-friendly and the process of adsorption was achieved artlessly with low cost manner.

The aim of this study was to investigate the capability of titanium dioxide nanoparticles, synthesized by sol gel method, to remove cationic Basic green-4 dye from aqueous solution. Basic green-4 generally used for dyeing of wool, jute and leather. It is highly cytotoxic mammalian cells and acts as a tumor enhancing agent. The kinetic and thermodynamic parameters of adsorption process was evaluated.

2. Materials and Methods

2.1. Materials

Titanium butoxide $[\text{Ti(IV)(O(CH}_2)_3\text{CH}_3)_4]$ (Sigma-Aldrich,97%), isopropyl alcohol (Roth,99%), hydrochloric acid (Sigma-Aldrich,37%), Basic green-4 (BG-4) (BDH,99%) with empirical formula of $\text{C}_{23}\text{H}_{25}\text{N}_2\text{Cl}$ and a molecular weight of 364.91 g/mol. All experimental solutions were prepared by using deionized water.

2.2. Methods

2.2.1. Preparation of TiO_2 Nanoparticles

The titanium dioxide nanoparticles (TiO_2 -nps) was prepared by a sol gel method, titanium butoxide used as a precursor and mixed with isopropyl alcohol in 1:4 volume ratio at room temperature. To this mixture, a solution of 1:1 H_2O :isopropyl alcohol was added drop wise under vigorous stirring. The colloidal suspension was adjusted to pH 3 by adding diluted solution of HCl and using pH-meter equipment. The resulting solution was stirred for 1hr. and then dried in an oven overnight at 70°C . The TiO_2 -nps powder was calcinated in a furnace at 500°C for 4hr.

2.2.2. Characterization of TiO_2 -nps

The TiO_2 -nps were characterized by FTIR spectrophotometer (Shimadzu,8500,Japan).XRD pattern of prepared nano TiO_2 was characterized by using XRD, Bruker AXS GmbH, Germany/D2 Phaser. Topography and morphology of the prepared sample were recorded by using Atomic Force Microscope (AA3000 Scanning Probe Microscope, Angstrom Advanced Inc.,USA) and Scanning Electron Microscope (JEOL JSM-6700 instrument ,Germany).

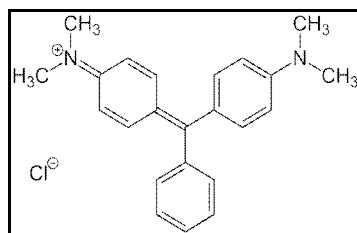
2.2.3. Adsorption Isotherm

Wavelength of maximum absorbancy (λ_{max}) was measured for BG-4 dye (Scheme1) and found 616nm (using UV-Visible spectrophotometer, Double beam, Shimadzu. PC 1650, Japan). This value was used to evaluate the quantity of dye adsorbed. The adsorption isotherms of BG-4 dye on TiO_2 -nps were performed by shaking 0.01g of surface into 10ml dye solutions with different initial concentrations ranging between 2-20 mg/L at constant temperature (30°C). After 1hr., the time that is sufficient to reach equilibrium, the TiO_2 -nps was separated by centrifugation at 6000 rpm for 15 min. This process was repeated to remove all fine particles of TiO_2 -nps from dye solution. The equilibrium concentration of dye was determined spectrophotometrically at 616 nm.

The quantity of dye adsorbed (q_e) was calculated according to the following equation:

$$q_e = (C_o - C_e)V / m \quad (1)$$

Where C_o and C_e (mg/L) are the initial and equilibrium concentrations of BG-4 dye. V (L) is the volume of solution and m (g) is the weight of TiO_2 -nps.



Scheme 1. The chemical structure of Basic Green-4

2.2.4. Kinetic and Thermodynamic Experiments

The kinetic studies was determined by adding 0.01g of TiO₂-nps into 10ml BG-4 dye solution with initial concentration 15 mg/L. The temperature of solution was held constant at 30°C with a thermostatic shaker (using shaker water bath, K&K, Scientific, Korea). The samples of reaction mixtures were withdraw at different time intervals and the residual BG-4 concentration was determined. The percentage of dye removal was calculated as follows:

$$\% \text{Removal} = ((C_0 - C_e) / C_0) \times 100 \quad (2)$$

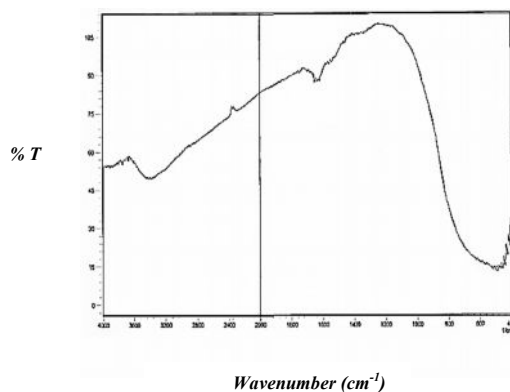
To estimate the thermodynamic functions of adsorption process, the adsorption experiment was repeated in the same manner at another two temperatures 40 and 50°C.

3. Results and Discussion

3.1. Characterization of TiO₂-nps

3.1.1. FTIR

FTIR spectrum of TiO₂-nps was shown in Figure 1. The broad absorption peaks around 3400 cm⁻¹ and 1640 cm⁻¹ in the spectrum corresponding to the stretching and bending vibration of hydroxyl group (-OH) in water. The broad peak at 480-560 cm⁻¹ refer to characteristic stretching vibration of Ti-O in titanium dioxide⁽³⁹⁾.

Figure 1. FTIR spectrum of prepared TiO₂-nps

3.1.2. X-ray diffraction (XRD)

The crystal size and phase composition of TiO₂-nps were estimated by X-ray analysis using CuK α radiation ($\lambda = 0.15406$ nm). The XRD pattern of the prepared TiO₂-nps was shown in Figure 2. The peaks at 2θ values 25.49, 37.98, 38.74, 48.23, 54.08, 55.3, and 68.9 corresponding to the Miller indices of (101), (004), (112), (200), (105), (211), and (116), respectively. By comparison the obtained data with that of standard JCPDS (JCPDS 21-1272) data, the prepared sample is predominantly composed of anatase crystalline phase of titanium dioxide.

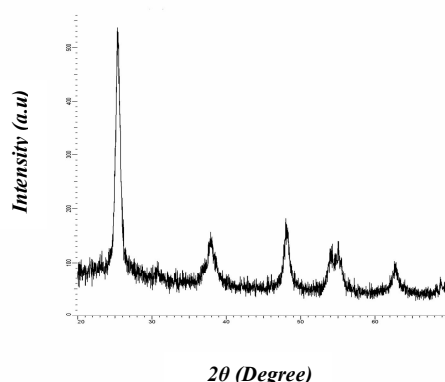


Figure 2. XRD pattern of the prepared TiO₂-nps

The average crystalline size of TiO₂-nps was estimated from Scherrer's equation⁽⁴⁰⁾:

$$D = K \lambda / \beta \cos \theta \quad (3)$$

Where D is the crystallite size, K is the Scherrer constant (usually taken as 0.94) for spherical shape, λ is the wavelength of the X-ray radiation, and β is the full width at half-maximum height, θ is the Bragg's diffraction angle.

The (101) diffraction peak for anatase phase was used to determine the value of crystal size of the prepared TiO₂-nps and it was found 9.92 nm.

3.1.3.AFM analysis

The surface topography of the prepared TiO₂-nps was analyzed using AFM spectroscopy. Figure 3 and 4 show 3D image and the granularity cumulation distribution chart of TiO₂-nps. The average diameter of TiO₂ particles is 108 nm. Its appear from AFM image that TiO₂ particles are randomly arranged and agglomerate to form larger particle size.

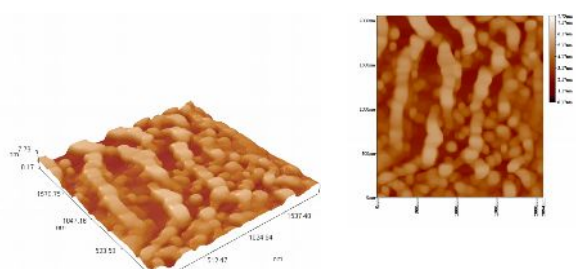


Figure 3. AFM 3D image of the prepared TiO₂-nps distribution

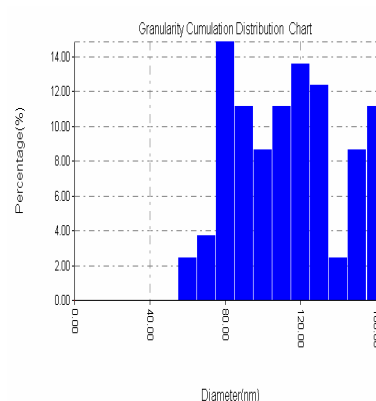


Figure 4. Granularity cumulation chart of TiO₂-nps.

3.1.4.SEM analysis

The surface morphology of the prepared TiO₂-nps was studied using SEM, as shown in Figure 5. The SEM image indicate that TiO₂ nanoparticles have almost spherical morphology and the agglomeration of nanoparticles was also significant.

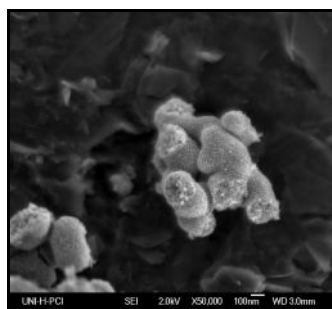


Figure 5. SEM image of TiO₂ powder

3.1.5. Adsorption of dye

The amount of BG-4 adsorbed on TiO₂-nps surface at 30 °C was represented in Figure 6. The adsorption capacity of TiO₂-nps surface increased as the initial dye concentration increased. The increase of dye concentration accelerates the mobility of dye molecules in the liquid phase, due to increase in the driving force of concentration gradient.

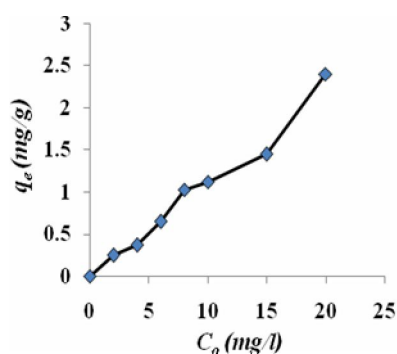


Figure 7. The effect of initial concentration on the amount adsorbed of BG-4 dye

The mechanism of BG-4 adsorption by TiO₂-nps was elucidated on the basis of FTIR analysis of surface loaded dye. Its indicated from Figure 8, that the intensity of peaks related to -OH and Ti-O groups were decreased after BG-4 adsorption and the broad peak of Ti-O group relatively shifted to higher wavenumber. This result may suggest the involvement of two groups in adsorption process of dye.

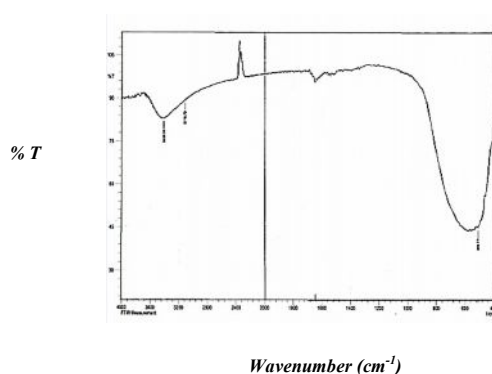


Figure 8. FTIR spectrum of TiO₂-nps loaded BG-4 dye

3.1.6. Adsorption isotherm

Adsorption isotherm is usually known as the relationships between the quantity of adsorption and the equilibrium concentration at fixed temperature. The normalized isotherm provide more physiochemical informations about the adsorption capacity of the solid surface and the net heat of adsorption process⁽⁴¹⁾. As shown in Figure 9, the characteristic type of BG-4 equilibrium isotherm is S₄-type according to Gile's classification⁽⁴²⁾.

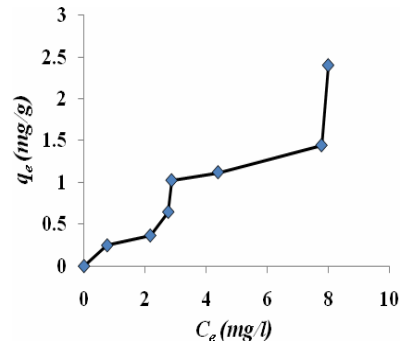


Figure 9. Adsorption isotherm of BG-4 on TiO₂-nps at 30 °C

The experimental adsorption data were tested for their applicability to Langmuir and Freundlich isotherm.

The low value of correlation coefficient of Langmuir isotherm ($r^2 = 0.095$), shows that the adsorption of BG-4 dye on TiO₂-nps follows isotherm which are well represented by applying the Freundlich model⁽⁴³⁾:

$$qe = K_f C_e^{1/n} \quad (4)$$

Where K_f and n are Freundlich constants. $1/n$ is a function of the strength of adsorption in adsorption process⁽⁴⁴⁾.

The logarithmic form is:

$$\log qe = \log K_f + \log C_e \quad (5)$$

The linear plot of $\log K_f$ versus $\log C_e$ (Figure 10) prove this fact. The values of correlation coefficient and Freundlich constants ($r^2 = 0.914$, $K_f = 9.418$, $n = 1.094$). The high n value indicating that the adsorption of BG-4 dye on the surface is favorable.

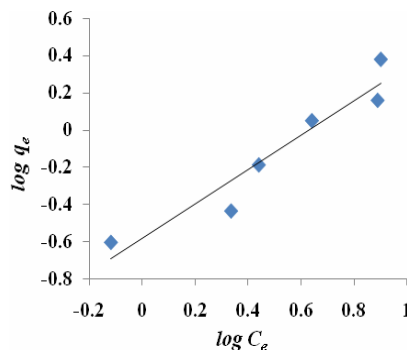


Figure 10. Linear form of Freundlich isotherm of BG-4 on TiO₂-nps

3.1.7. Thermodynamic parameters

The thermodynamic treatment of adsorption enables informations to be obtained about the magnitude of the bond strength, randomness and spontaneity of the adsorption process. As shown from Figure 11, the adsorption uptake of dye on TiO₂-nps was decreased with the rise in temperature from 30 to 50 °C. This result could be interpreted as a weakening of attractive forces between BG-4 molecules and the surface with the increase of temperature.

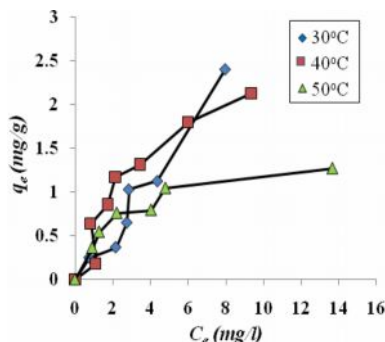


Figure (5). Adsorption isotherms of BG-4 on TiO₂-nps at different temperatures

One of the foremost tasks of adsorption thermodynamics is the analysis of heat effects which accompany adsorption. The heat of adsorption could be determined using Van't Hoff equation:

$$\ln X_m = \frac{-\Delta H}{RT} + \text{constant} \quad (6)$$

Where X_m is the maximum amount adsorbed at a certain value of concentration equilibrium.

The other thermodynamic parameters (ΔG, ΔS and K) are calculated based on equation:

$$\Delta G = RT \ln K \quad (7)$$

$$\Delta G = \Delta H - T \Delta S \quad (8)$$

The negative value of enthalpy (ΔH= -27.203 kJ/mol) and entropy (ΔS= -9.978 J.mol⁻¹.K⁻¹), confirmed the exothermic process and that the BG-4 molecules have ordered arrangement on TiO₂-nps surface. On the other hand the positive and low value of free energy and equilibrium adsorption constant (ΔG= +3.032 kJ/mol, K= 0.3) revealed that the adsorption process was thermodynamically non-spontaneous in nature.

3.1.8.Kinetic analysis

Figure 12 showed rapid percentage removal of dye by TiO₂-nps in the first few minutes, nearly 40% of BG-4 dye was removed from an aqueous solution within 60 min.

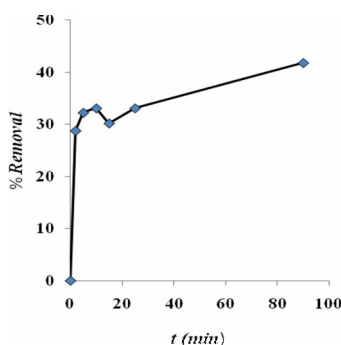


Figure 11. Effect of contact time on the removal of dye

The experimental kinetic data were analyzed according to pseudo-first order and pseudo-second order kinetic models.

The linear form of pseudo-first order equation⁽⁴⁵⁾ is expressed as:

$$\ln (q_e - q_t) = \ln q_e - k_1 t \quad (9)$$

Where q_e and q_t are amount of dye adsorbed (BG-4/g) at equilibrium and time t(min.) respectively, k₁ is the rate constant of pseudo-first order kinetic.

The low value of correlation coefficient ($r^2 = 0.259$) of the plot of $\ln(q_e - q_t)$ vs. t provided that pseudo-first order adsorption model is less suitable to describe the adsorption kinetic of BG-4 dye on the solid surface.

The pseudo-second order kinetic model⁽⁴⁶⁾ is:

$$t/q_t = 1/h + (1/q_e) t \quad (8)$$

Where h is the initial adsorption rate and is equal to: $h = k_2 q_e^2$

k_2 is the pseudo-second order rate constant.

The linearity of the plot t/q_t vs. t (Figure 12) with good correlation coefficient ($r^2 = 0.993$) indicates the applicability of the second-order model to the adsorption of dye on solid surface. In addition the theoretical q_e value ($q_e = 1.288$) calculated by this kinetic model was close to that found by experiment.

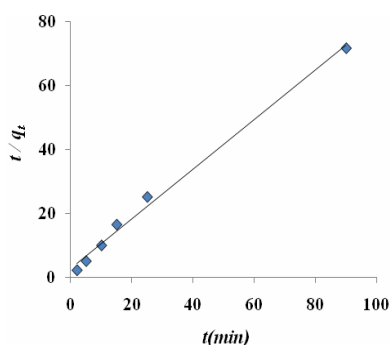


Figure 12. Applicability of pseudo-second order kinetic model to dye adsorption

The experimental data were also analyzed by intraparticle diffusion kinetics model⁽⁴⁷⁾ to investigate the rate controlling step:

$$q_t = k_d t^{1/2} + C \quad (9)$$

Where k_d ($mg \cdot g^{-1} \cdot min^{-1/2}$) is the intraparticle rate constant and C (mg/g) is a constant. The plot of q_t versus $t^{1/2}$ was shown in Figure 13 and the values of k_d , C and r^2 are 0.043 , 0.810 and 0.859 respectively. The straight line of intraparticle diffusion equation don't pass through the origin point, this result demonstrated that intraparticle diffusion may not be the controlling step in the adsorption of BG-4 dye on TiO_2 -nps surface⁽⁴⁸⁾.

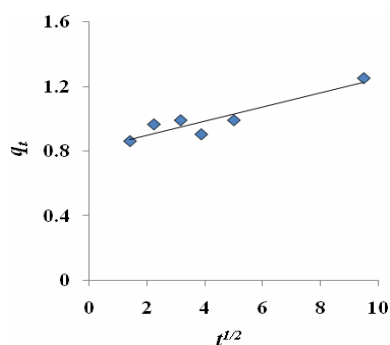


Figure 13. Applicability of intraparticle diffusion kinetic model to dye adsorption

Conclusions

The TiO_2 -nps were prepared by sol gel method. The XRD, AFM and SEM analysis of synthesized sample indicated that TiO_2 -nps have spherical shapes with diameter sizes approximately 108.3 nm. The adsorption experiments showed that the synthesized adsorbent revealed relatively high adsorption efficiency of BG-4 dye. The adsorption process follows Freundlich isotherm and pseudo-second order kinetic model. The

thermodynamic analysis of adsorption data showed that the removal of dye was endothermic and non-spontaneous.

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