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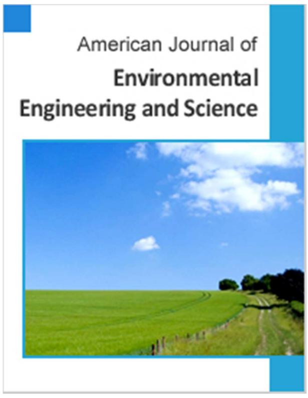

Adsorption of Eriochrom Black T Azo Dye onto Nanosized Anatase TiO$_2$

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Abstract
Nanosized anatase TiO$_2$ powder was used for the adsorption of Eriochrome Black-T (EBT) azo dye from aqueous solution. The impact of the operational parameters namely; pH, contact time, initial dye concentration, temperature and adsorbent dose were studied using UV–Visible spectroscopic technique at wavelength = 540 nm. The adsorption kinetics were described better using pseudo-second order model and the adsorption isotherm was elucidated via Langmuir and Freundlich models which indicate that the EBT adsorption onto anatase TiO$_2$ nano catalyst fitted with both models, however better with Freundlich isotherm. The equilibrium time was found to be 30 minutes. The best results were achieved at pH = 3 and T = 293 K. The thermodynamic parameters, ΔG, ΔH, and ΔS were calculated. The negative value of ΔH indicates that the adsorption process is exothermic.

1. Introduction

Dyes have extensively been used for long time in several industries like; dyeing, paper, pulp, textiles, plastics, leather, paint, cosmetics and food industries. About 10-15% of dyes are being wasted into the environment after the completion of dyeing process [1]. A considerable percentage of these dyes are discharged into the effluent water as they are highly water solvable [2]. The dyes could cause health problems due to their toxic, mutagenic and carcinogenic characteristics. Moreover, they are very stable to light, temperature and microbial attack, making them recalcitrant compounds [3,4]. The waste water that contains heavy metals, chemicals, dyes, oils and many other harmful materials are discharged by the industries into the water bodies without suitable treatment, which leads to pollution of water resources [5]. Therefore, it is extremely important to get rid of these pollutants from waste water before final disposal. Among several chemical and physical methods, adsorption process is one of the effective techniques that have been successfully employed for removing color from waste water. The largest class of dyes used in textile processing industries are almost azo compounds i.e. molecules with one or more azo (N=N) bridges linking substituted aromatic structures [4]. Eriochrome Black T (EBT) dye has also been used as an indicator in complexometric titrations for determination of Ca^{2+}, Mg^{2+} and Zn^{2+} ions and for biological staining. This dye is dangerous as such and its degradation products like naphtaquinones are still more carcinogenic. A literature survey showed that only few papers have studied the removal of EBT [6]. TiO$_2$ is used as a tool for the adsorption of dyes including EBT for photocatalysis purpose due to its long-term stability, low cost, availability and non-toxicity [7]. The present work focuses on the study of the adsorption of EBT by anatase TiO$_2$ nanoparticles, as a prerequisite for the photodegradation process, encompassing the impacts of several experimental parameters which govern the feasibility of the process like initial pH, contact time, initial dye concentration, temperature, and adsorbent loading. In addition the kinetic analysis was conducted to investigate the mechanism of dye adsorption.

2. Experimental

2.1. Material

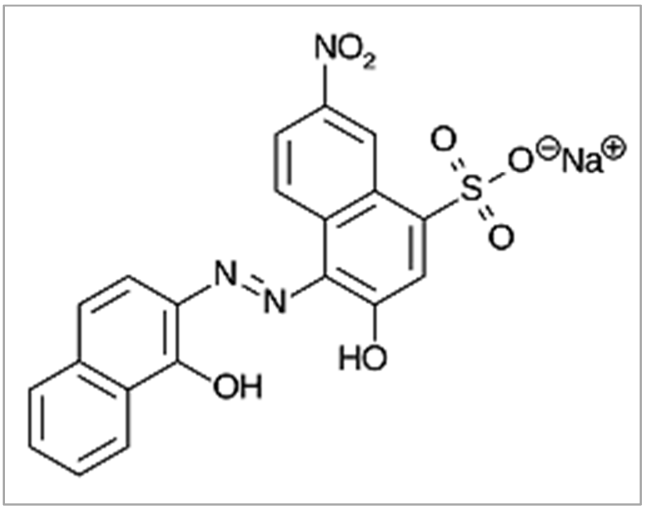

Fig. 1. The chemical formula of EBT azo dye (1-[1-hydroxy-2-naphthol azo]-6-nitro -2-Naphthol- 4-sulphonic acid sodium salt).

The anionic dye Eriochrome Black T (EBT) as adsorbate (Fig. 1) in commercial purity was used (>99.5%). Nanosized TiO_2 (25 nm) powder as adsorbent was purchased from Aldrich with purity of 99.7%. All other reagents were analytical grade and used without further purification.

2.2. Apparatus

Apel PD-303 single beam visible spectrophotometer, pH multimeter (HANNA instruments) and Centrifuge (K centrifuge PLC series.) were used throughout this work.

2.3. Batch Mode Adsorption Methodology

Batch adsorption experiments have been performed to explore the impacts of initial solution pH, adsorbent loading, initial dye concentration and contact time. The stock solution of 500 mg EBT L^{-1} was prepared by dissolving an accurately weighed quantity 0.5 g of solid EBT dye in 1 L of deionized water. The experiments' solutions of desired concentrations (10-100 mg L^{-1}) were prepared by further dilutions of accurate proportions of stock solution into 100 ml volumetric flasks. The pH of the experiments' solutions was adjusted using 0.1 M HCl or 0.1 M NaOH. The adsorption experiments were conducted by adding 50 mL of dye solution onto 0.1 g of adsorbent (anatase TiO_2) in 100 ml round flask and the mixture was agitated at room temperature using a mechanical shaker and at a speed of 100 rpm. The adsorption process was monitored by determining the absorbance and concentration of EBT in the processed solution using single beam visible spectrophotometer and at λ_{max} = 540 nm. The adsorbed amounts of EBT dye were calculated from the equilibrium concentrations after adsorption according to expression (1);

$$Q_e = V_{sol.} (C_o - C_e) / m \quad (1)$$

Where C_o and C_e are the initial and equilibrium liquid phase concentrations of dye (mg L^{-1}), respectively; Q_e is equilibrium dye concentration on adsorbent (TiO_2) (mg g^{-1}). V is the volume of dye solution (L), and m is the mass of nanosized TiO_2 used (g). Furthermore, the percentage of EBT adsorption (% ads.) on anatase TiO_2 was calculated using equation (2);

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

2.4. Calibration Curve

The calibration curve was drawn between the absorbance values of the diluted stock solutions (10-100 mg L^{-1}) which were measured at optimal wavelength (λ_{max} = 540 nm) and then plotted against the corresponding concentrations. Figure 2 shows the calibration curve in the concentration range that falls in the region of applicability of Beer-Lamberts law.

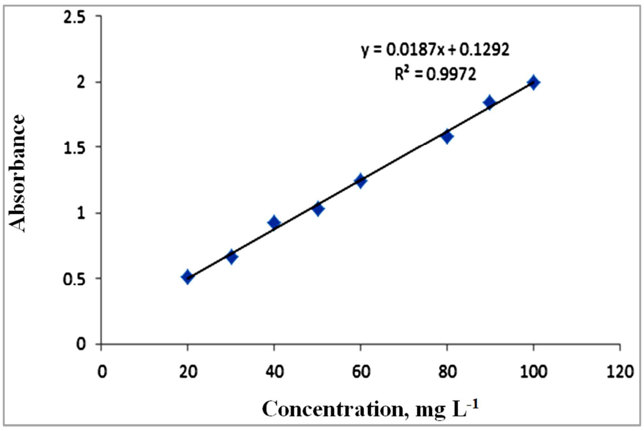

Fig. 2. Calibration curve of EBT.

3. Results and Discussion

3.1. Effect of Contact Time

The effect of contact time on the adsorbed amount of EBT dye per unit of adsorbent was investigated at room temperature with constant pH and dye concentration. Figure 3 and Table 1 show the variation of Q_e and C_e of 50 ppm EBT at 298K and pH = 5.

The dye adsorption increased significantly as the shaking time increased up to 15 minutes with a quick uptake of the dye. The adsorption rate however decreased to a stable value with increase in contact time due to probably a shortage of available adsorption sites for binding [6]. However, a maximum value of adsorption is attained after 30 minutes of shaking time. Therefore, the time of 15-30 minutes may be treated as the optimum contact time and accordingly was used for the following adsorption experiments.

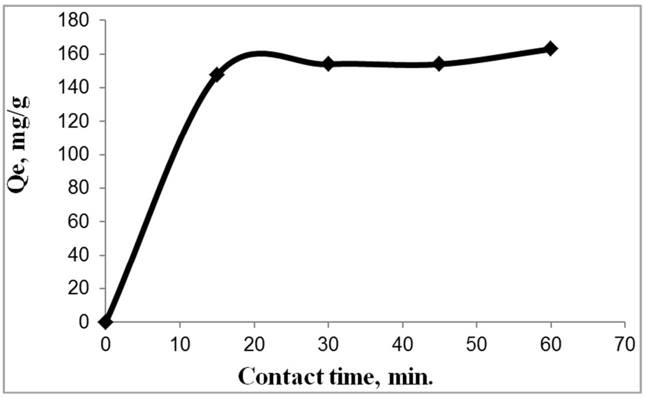

Fig. 3. *The variation of Q_e with contact time of 50 mg EBT L^{-1} solution at 298K temperature and pH = 5.*

Table 1. *The values of Q_e and C_e at different time of adsorption for 50ppm of EBT solution at 298K temperature.*

Time, min	C_e,mg/l	Q_e,mg/g
15	41.5	147.3
30	40.8	153.9
45	40.8	153.9
60	39.8	162.9

3.2. The Effect of pH

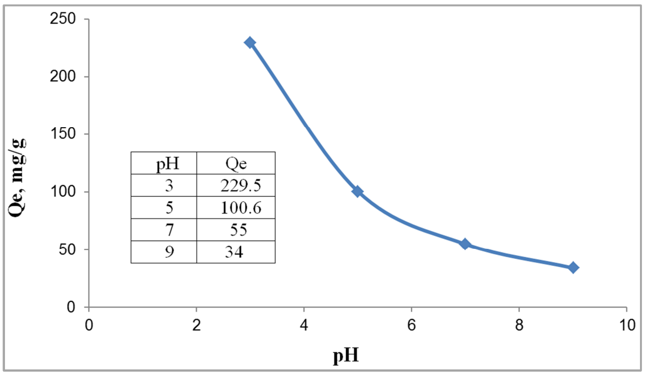

Fig. 4. *Effect of initial pH on the sorption of EBT on TiO_2.*

The pH of dye solution has been identified as one of the significant parameter because it plays an important role in the whole adsorption process and particularly on the adsorption capacity [8]. Figure 4 depicts the influence of solution initial pH on the sorption of EBT. The activity of adsorption depends on the solution pH, since variation in pH leads to the variation in the degree of ionization of the adsorptive molecule and the surface properties of adsorbent [9]. From Fig. 4 we observe that the sorption of EBT dye attained the maximum at the initial pH = 3 and decreased with increasing the pH within the investigation range of 5–9. At alkaline pH, two governing factors play a significant role, first, lower adsorption of anionic dye, EBT, due to the presence of excess of $^{-}$OH ions which competing with the dye anions for the adsorption sites [10], and on the other hand, TiO_2 is negatively charged at higher pH, which also contributes in the electrostatic repulsion between the negatively charged dye molecule and the negatively charged adsorbent [1]. Whereas, as the pH of the system decreases, the number of positively charged surface sites on the adsorbent increases and consequently favors the adsorption of the anions due to Columbic electrostatic attraction [10]. One could also report that at high pH the sulphonic acid group is completely deprotonated which make the dye molecule more soluble in water, so the adsorption decreased with increasing pH.

3.3. Effect of Adsorbent Loading

The adsorbent loading determines the capacity of the adsorbent for a given initial dye concentration. Figure 5 illustrates the impact of adsorbent loading on the removal of 50 mg L^{-1} EBT dye. The dye removal percentage extends as the adsorbent dose increases from 0.1 to 0.4 g. At a low loading of 0.1 g TiO_2, there is firm competition between the EBT molecules due to the limited number of available binding sites; hence a low sorption was achieved. An increase in adsorbent dose to 0.4 g certainly causes a corresponding increase in removal yield due to higher active adsorption sites [11].

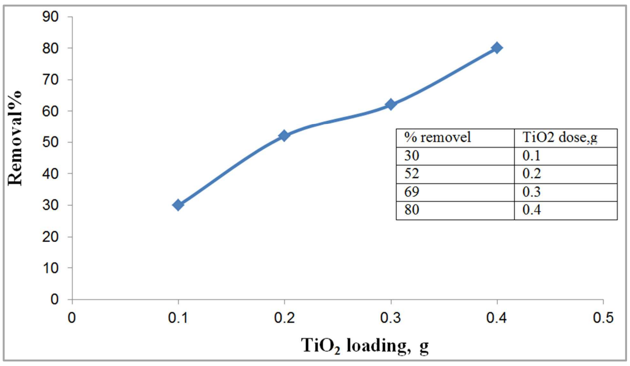

Fig. 5. *Effect of adsorbent loading on EBT removal.*

3.4. Effect of Initial Dye Concentration

The effect of initial dye concentration depends on the immediate relation between the concentration of the dye and the available sites on an adsorbent surface [12]. From Fig. 6 we observe that the adsorption capacity Q_e increases with increasing the initial dye concentration until attains the maximum at Q_e = 143 mg/g, and then starts decreasing. This is due to saturation of adsorption sites of the adsorbent material [13]. Hence, the amount of adsorbed EBT is highly dependent on the initial dye concentration.

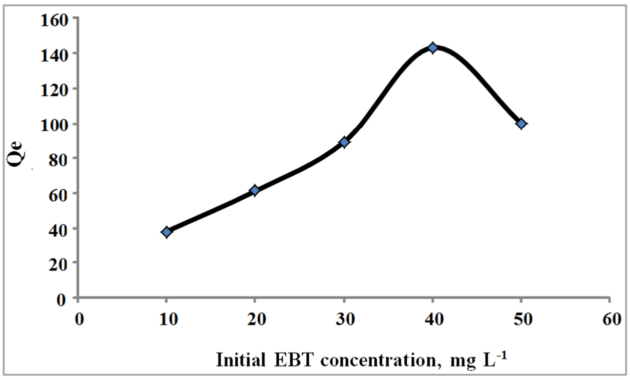

Fig. 6. *Impact of initial dye concentration on the adsorption of EBT onto TiO_2.*

3.5. Effect of Temperature

The adsorption process temperature is another important physico-chemical operational parameter, because it contributes massively in variation of adsorption ability of the adsorbent [12]. The adsorption experiments of EBT onto nanosized TiO_2 were carried out at three different temperatures namely; 30, 40 and 50°C with an accuracy of ±0.5 ºC. It is known that the process of adsorption is often an exothermic in nature, thus a decrease in temperature of the system would result in an increase of adsorption and vice verse [13-15]. Figure 7 corroborates the effect of the process temperature on the adsorption efficiency at TiO_2 dosage of 10 mg, agitation time of 30 min and 40 mg L^{-1} EBT concentrations. In chemisorption, the quantity adsorbed may increase or decrease with the rising temperature depending on the type of interaction and bonding between the adsorbate molecules and the adsorbent surface, while in physisorption a decrease in temperature enhances the extent of adsorption [14]. Also from Fig. 7, a decrease of adsorption ability with increasing temperature verifies the exothermic feature of the adsorption process. Because higher temperature induces higher mobility of the adsorbate causing desorption [15].

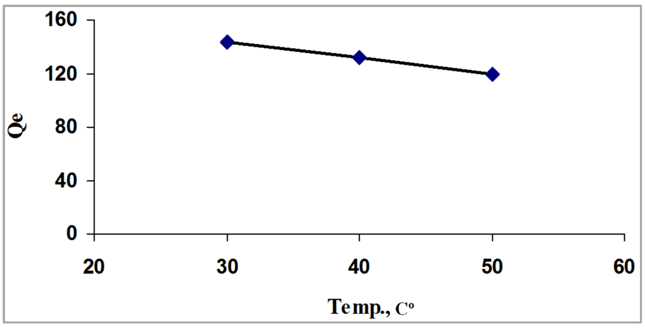

Fig. 7. *Effect of temperature on the adsorption of 40 mg L^{-1} EBT onto TiO_2.*

3.6. Thermodynamic Parameters

In order to evaluate the present adsorption process, certain thermodynamic factors were computed, because the thermodynamic functions contribute essentially in understanding the nature of adsorption. The thermodynamic parameters which are related generally to the adsorption of dyes, such as, enthalpy change ΔH^o (kJ/mol), entropy change ΔS^o (J/ mol. K) and Gibbs free energy change ΔG^o (kJ/mol) have been calculated according to the following expressions:

$$K_p = Q_e / C_e \tag{3}$$

$$\Delta G = -R\,T \ln K_p \tag{4}$$

$$\ln K_p = -\Delta H^o / RT + \Delta S^o / R \tag{5}$$

Where K_p is the thermodynamic equilibrium constant, i.e., the ratio of the equilibrium concentration of EBT on TiO_2 (Q_e) to that in solution (C_e). T is the absolute temperature in Kelvin, and R is the universal gas constant (8.314 J/mol. K). ΔH^o has been calculated according to Van't Hoff equation (eq. 5) via plotting logarithmic value of the thermodynamic equilibrium constant (K_p) against the reciprocal of absolute temperature as (1/T). The Plotting of $\ln K_p$ against 1/T gives a straight line with slope and intercept equal to $-\Delta H^o/R$ and $\Delta S^o/R$, respectively (Fig. 8). A linear fit with an equation of $\ln K_p = {}^{-}1900.3/T + 5.4716$ was obtained in this experiment and the values of ΔH^o and ΔS^o calculated from Fig. 8 were -15.79 kJ/mol and 45.49 J / mol. K, respectively.

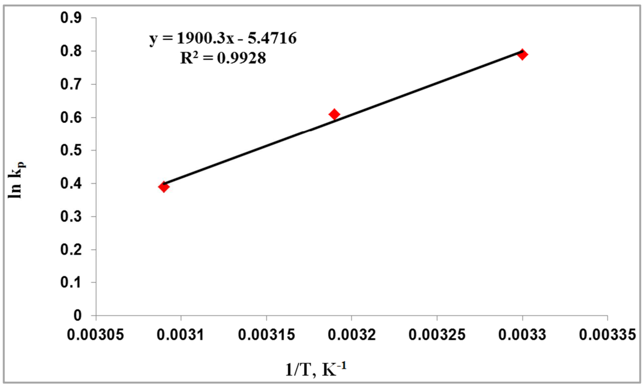

Fig. 8. *Van't Hoff plot for adsorption of EBT on TiO_2.*

The values of thermodynamic parameters are presented in Table 2. The ΔH° negative value indicates that the adsorption of EBT dye onto nanosized TiO_2 has been an exothermic process, which was supported by the increase of adsorption of the dye with decrease of temperature (Fig. 7). The ΔS° positive value indicates that the degrees of randomness (freedom) increased at the solid – liquid interface during adsorption of EBT [15, 9]. Also from Table 2, we report here that the negative ΔG^o values indicate the feasibility of the EBT adsorption process onto solid TiO_2 surface in addition to the spontaneous nature at all the studied temperatures. This conclusion is well concerted with other authors [16, 2]. Hence, we conclude that the adsorption of EBT onto nanosized TiO_2 is spontaneous and thermodynamically favourable. On the other hand, some authors [15-16] found that the ΔG° value for physisorption process is between 0 and -20 kJ/mol, whereas it could attain a value of -400 kJ/mol for chemisorption process.

Table 2. *Thermodynamic functions of the adsorption process.*

Temp., K	ΔG, kJ/mol	ΔH, kJ/mol	ΔS, $JK^{-1}mol^{-1}$
303	-1.99	-15.79	45.49
313	-1.58	-	-
323	-1.04	-	-

3.7. Adsorption Kinetics

In this work, pseudo-first-order and pseudo-second-order kinetics model were applied to examine the controlling mechanism of EBT adsorption onto nanosized TiO_2 from aqueous solutions. The rate of adsorption of EBT has been studied as a function of time as it is depicted in Fig. 3. The adsorption equilibrium was reached in approximately 30 minutes. Both the first and the second-order rate equations were used to determine the most suitable rate expression for adsorption of the investigated dye on TiO_2. The optimal model was considered based on correlation coefficient (R^2).The correlation coefficient was frequently used to decide whether the model represents correctly the experimental data.

3.7.1. The First-Order Kinetics Model

The rate constant of adsorption is determined from the first-order rate expression given by the famous linearized form of Lagergren rate equation [17] as follows:

$$\ln (q_e - q_t) = \ln q_e - k_1 t \qquad (6)$$

where q_t and q_e (mg. g^{-1}) are the amount of material adsorbed (adsorption capacity) at time t and at equilibrium, respectively. k_1 (min^{-1}) represents the rate constant of the first-order model.

3.7.2. The Pseudo-Second-Order Kinetics Model

The pseudo-second-order equation was defined as follows [18]:

$$t/q_t = 1/(k_2 q_e^2) + t/q_e \qquad (7)$$

where k_2 (g. mg^{-1} min^{-1}) is the rate constant of the second-order model.

The kinetics parameters for adsorption of EBT under different conditions were calculated from equations 6 and 7 and are given in Table 3. The correlation regression coefficient (R^2), for the first-order kinetic model is 0.75 while the correlation regression coefficient (R^2), for the pseudo-second-order kinetic model is 0.995 (figures were not shown). Calculated correlation regression coefficient (R^2) is closer to unity for second-order kinetics model; therefore the adsorption kinetics could well be approximated more favorably by second order kinetics model for EBT adsorption. Based on the pseudo-second-order model, these observations showed that the value of dye adsorption process was controlled by the chemisorption process, which depends on the chemical characters of both TiO_2 and dye [16]. Azizian [19] explored the kinetics of adsorption from a solution onto an adsorbent theoretically, and found that the adsorption process obeyed first-order kinetics at high initial concentration of solution while it obeyed pseudo-second-order kinetics at lower initial concentration of solution.

Table 3. *Comparison of the first and second-order adsorption rate constants for 50 mg L^{-1} EBT adsorption onto TiO_2.*

Time	Fisrt order kinetics model					Second order kinetics model		
	R^2	k_1	q_e	q_t	$\ln q_e - q_t$	R^2	K_2	t/q_t
15	0.75	0.0180	162.9	147.3	2.747	0.99	0.0058	0.108
30	-	-	-	153.9	2.197	-	-	0.19
45	-	-	-	153.9	2.197	-	-	0.29
60	-	-	-	162.9	-	-	-	0.36

3.8. Adsorption Isotherm Models

The adsorption isotherms play an important role in elucidating the interaction between the adsorbent and the adsorbate, and further deepen the understanding of the mechanism of adsorption phenomenon. Several isotherm models are presented in the literature [20]. Langmuir and Freundlich models are the most widely used to describe the adsorption isotherm. The Langmuir model assumes that adsorption takes place at uniform energy sites on the surface of the adsorbent. The linearized Langmuir isotherm equation can be expressed as follows:

$$C_e/Q_e = C_e/Q_{max} + 1/K_L Q_{max} \qquad (8)$$

where C_e is the equilibrium concentration of EBT in solution (mg/L), Q_e is the amount of dye adsorbed on adsorbent (mg/g) at equilibrium, Q_{max} is the maximum adsorption capacity (mg/g), and K_L is the Langmuir binding constant, which is related to the energy of adsorption (L/mg). The slope and intercept of C_e / Q_e vs. C_e plot were utilized to calculate $1/Q_{max}$ and $1/(K_L Q_{max})$ respectively, as exhibited in Fig. 9.

The Freundlich isotherm, Fig. 10, is an empirical equation based on adsorption on a heterogeneous surface. The equation is commonly represented by:

$$\text{Log } Q_e = \log K_f + \log C_e/n \qquad (9)$$

Where K_f and n are the Freundlich constants as characteristics of the system, indicating the adsorption capacity and the adsorption intensity, respectively. When the value of 1/n is lower than 1 (n > 1), it indicates a normal Freundlich isotherm, otherwise, it is indicative of cooperative adsorption.

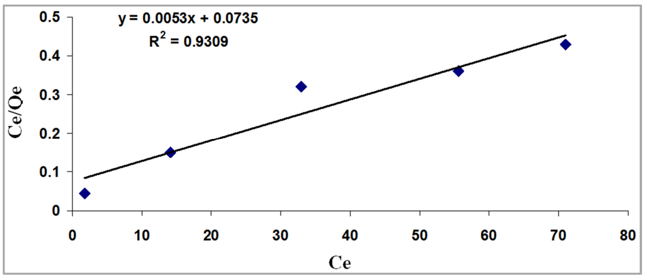

Fig. 9. *The linear plot of Langmuir isotherm for adsorption of EBT onto TiO_2*

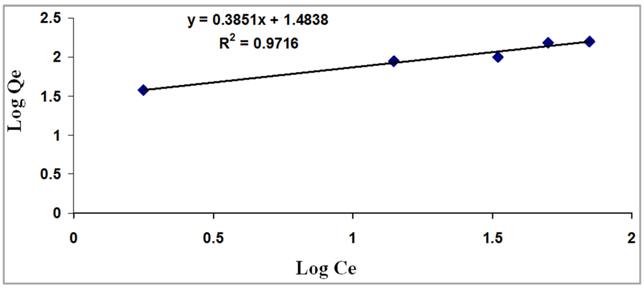

Fig. 10. *The linear plot of Freundlich isotherm for adsorption of EBT onto TiO_2.*

The calculated parameters of both Langmuir and Freundlich models which resulted upon the correlation of adsorption data for different initial EBT concentrations are presented in Table 4. The comparison of correlation coefficients (R^2) for Figs. 9 and 10 refers to higher R^2 value possessed by Freundlich model. This suggests that this model yields a better fit in describing the experimental equilibrium adsorption of EBT onto TiO_2. Hence, The Freundlich isotherm is an empirical equation that assumes heterogeneous adsorbent surface with its adsorption sites at varying energy levels [21]. Moreover, the maximum adsorption capacity, Q_{max}, of the process of this work is compared in Table 5 with the data reported by other authors for EBT adsorption.

Table 4. *Adsorption isotherms parameters for EBT adsorption onto* TiO_2*.*

Freundlich isotherm			**Langmuir isotherm**		
1/n	K_f	R^2	Q_{max}	K_L	R^2
0.38	1.48	0.971	188.6	0.072	0.931

Table 5. *Maximum adsorption capacities of EBT from aqueous media onto various adsorbents*

Adsorbent	**Max. Capacity, Q_{max}, mg/g**	**Reference**
Nano TiO_2 (this work)	188.6	-
Activated carbon	160.4	11
Eucalyptus bark	52.4	2
$NiFe_2O_4$ nanoparticles	47	6

4. Conclusions

Thermodynamic study indicated that the adsorption of EBT onto TiO_2 surface is spontaneous and exothermic as evidenced by negative values of the changes in Gibbs free energy (ΔG) and enthalpy (ΔH). Accordingly, the exothermic nature of the process is approved as the amount of EBT dye adsorbed onto the adsorbent decreases with an increase in the adsorption temperature of the dye solution from 30-50°C.

The pseudo-second order equation best describes the kinetics of the EBT/TiO_2 adsorption system due to its higher R^2 value than that of the pseudo-first order. The Freundlich isotherm best described the equilibrium data with $R^2 = 0.971$, which signifies that a heterogeneous adsorption takes place between EBT dye and TiO_2. Conclusively, TiO_2 is an effective adsorbent for the sorption of EBT dye from aqueous solution, where process parameters such as solution pH, initial dye concentration and adsorbent dose significantly affect the % EBT sorption.

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