

Application of Modified Bagasse as a Biosorbent for Reactive Dyes Removal from Industrial Wastewater

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ABSTRACT

Propionic acid modified bagasse was used for the removal of reactive yellow 2 and reactive blue 4. The effects of pH, contact time, initial dye concentrations, adsorbent particle size and adsorbent dose on the adsorption of the two dyes were investigated. Additionally, the desorption process and intra-particle diffusion were studied. Acidic pH values were favorable for adsorption of both dyes. The equilibrium adsorption data were best fitted with the Freundlich isotherm for reactive yellow 2 and the Langmuir isotherm for reactive blue 4. The values of their corresponding constants were determined. The kinetic for dye adsorption is well described by a pseudo-first order kinetic model for the reactive yellow 2 and by pseudo-second order for the reactive blue 4. The investigation revealed that the hydroxyl groups of bagasse and the carboxylic group of propionic acid play a great role in the removal of both reactive dyes.

Keywords: Modified Bagasse; Reactive Yellow 2; Reactive Blue 4; Adsorption; Kinetics

1. Introduction

Dyes are widely used in many industries such as textile, plastic, paper, food and cosmetic for coloring their products. These dyes are water pollutants and cause environmental hazards. In addition, many dyes are difficult to be biodegradable and tend to persist in the environment creating serious water quality and public health problems. So, a conventional biological treatment process is not very effective in treating dyes wastewater. There are various methods for removing dyes including coagulation and flocculation [1], oxidation or ozonation [2] and membrane separation [3]. However, these processes are very expensive and cannot be effectively used to treat the wide range of dyes waste. Alternative methods include adsorption on natural biosorbents which are becoming now of high interest for removal of dyes from waste effluents. In this way several studies have used natural materials locally available, renewable and of low costs [4-6]. Among these sorbents are banana, bagasse, coir pith, palm-fruit bunch, apple-pomace, wheat straw, rice husk, pine sawdust, peanut hull, coffee husk, maize cob, orange peel, sugar cane dust and luffa fiber [7-9].

Recently, sugarcane bagasse was used as potential low-cost adsorbent for removal of many dyes from wastewater [10-14].

Reactive dyes are generally used for colouring cotton and other cellulosic material, but are also applied to a small extent on wool and nylon. In the present study removal of reactive yellow 2 and reactive blue 4 from aqueous solutions using bagasse pre-treated with propionic acid was investigated. Several parameters have been studied such as pH, contact time, biomass dosage, and particle size and desorption process. The equilibrium sorption capacity and the kinetic adsorption of both dyes on the modified bagasse were studied. The selection of bagasse for this study was based on low cost raw material and environmental impact.

2. Materials and Methods

Reactive yellow 2 and reactive blue 4 dyes were purchased from Sigma-Aldrich Chemical Co. The chemical structures of the two dyes are presented in **Figure 1**.

2.1. Preparation of Adsorbent

Sugar cane bagasse modified with propionic acid was previously described [13].

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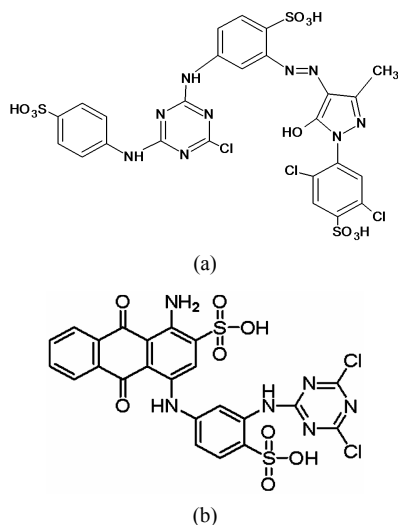


Figure 1. Chemical structures of reactive yellow 2(a) and reactive blue 4(b).

2.2. Preparation of Aqueous Dye Solution

Stock solutions of the two dyes were prepared by dissolving accurately a weighed amount of each dye in distilled water (1000 mg/L) and diluting these solutions prior to performing the adsorption experiments.

2.3. Adsorption Experiment

In each adsorption experiment 50 mL of dye solution of known concentration and pH was added to known weight of adsorbent in 100 mL airtight volumetric flasks at room temperature ($27^{\circ}\text{C} \pm 1^{\circ}\text{C}$) and the mixture was stirred on a rotary orbital Shaker at 150 rpm. The dye solution was filtered from bagasse and determined spectrophotometrically by recording the absorbance changes at maximum absorption (405 nm for reactive yellow 2 and 595 nm for reactive blue 4) using Thermofischer Scientific, aquamate, Evolution 166 Uv-visible spectrophotometer. All pH measurements were carried out with a pH-meter model number JENWAY 3450. The initial pH values were adjusted with 0.1 M HCl or NaOH to form series of pH's. Effect of adsorbent dosage was studied with different adsorbent doses and 50 ml of the dye solution at equilibrium time. For the desorption studies the adsorbent used for the adsorption of the dye solution was thoroughly washed with distilled water to remove unadsorbed dye and desorption process was conducted at a range of pH values. The amount of dye adsorbed onto the bagasse, q_e (mg/g) was calculated as the following equation:

$$q_e = (C_i - C_e)V/W \quad (1)$$

where C_i and C_e are the initial and equilibrium time solution concentration of the dye (mg/L), respectively, V the volume of the solution (L) and W the dry weight of the

sorbent (g).

2.4. Kinetic Studies

To assess the applicability of the adsorption process it is necessary to determine the kinetic parameters, using the batch technique. A series of volumetric flasks containing 100 mL capacity, containing 50 mL dye solutions of known concentrations at the appropriate pH and were agitated by mechanical shaker at room temperature. After a definite time interval, the solutions were filtered and the filtrates thus obtained were then analyzed spectrophotometrically. All the experiments were duplicated and only the mean values are reported. The maximum deviation observed was less than $\pm 5\%$.

2.5. Thermal Studies

Thermogravimetric analysis TGA and differential thermal analysis DTA techniques of original and treated with propionic acid (5 wt%) were carried out using a Shimadzu Thermal Analyzer model (TG 60 H). Curves of TGA and DTA were recorded upon heating up to 600°C at $10^{\circ}\text{C}\cdot\text{min}^{-1}$ and flow of air of $30\text{ mL}\cdot\text{min}^{-1}$.

3. Results and Discussion

3.1. Characteristics of Dyes

Table 1 summarizes some important characteristics of reactive yellow 2 and reactive blue 4 dyes.

3.2. Thermal Analysis

Figure 2(a) shows that the decomposition of untreated bagasse proceeds in five stages. The first region of ($36^{\circ}\text{C} - 124^{\circ}\text{C}$) which accompanied with 5.3% weight loss with an endothermic peak minimized at 77°C is attributed to the physically adsorbed water. The second stage of ($125^{\circ}\text{C} - 208^{\circ}\text{C}$) which accompanied with 8.1% weight loss is due to the dehydration water. The major weight loss (53.2%) takes place within ($209^{\circ}\text{C} - 358^{\circ}\text{C}$) range which exhibit exothermic peak maximized at 316°C . This peak is corresponded to the decomposition of cellulose and hemicelluloses in bagasse [14]. The fourth stage of ($359^{\circ}\text{C} - 459^{\circ}\text{C}$) and with exothermic peak maximized at 426°C and the fifth region of ($462^{\circ}\text{C} - 600^{\circ}\text{C}$) which accompanied with 4% weight loss with exothermic peak maximized at 480°C are attributed to the decomposition of lignin component of bagasse [14]. The TGA and DTA curves of treated bagasse with 5 wt% propionic acid was carried out and presented in **Figure 2(b)** and the data are cited in **Table 2**, it shows that the thermal decomposition of treated bagasses proceeds in similar behaviour of untreated bagasse *i.e.*, takes place in five stages. It is clear from the DTA curve and the data depicted in **Table 2** that the addition of propionic acid retards the third decompo-

Table 1. Characteristics of the used reactive dyes.

Dye	λ_{max} (nm)	C(wt%)	No. of ionizable groups	No. of functional groups available for interaction				
				-SO	-C=O	-NH	-Cl	-NH ₂
Reactive yellow 2	404	34.5	3	9	1	2	3	-
Reactive blue 4	595	35	2	6	2	2	2	1

Table 2. Data for thermal decomposition of untreated and treated bagasse.

Adsorbent	Temperature range (°C) and weight loss (%)					Total weight loss (%)
	1 st stage	2 nd stage	3 rd stage	4 th stage	5 th stage	
Original bagasse	36 - 124	125 - 208	209 - 358	359 - 459	462 - 600	91.7
	5.3	8.1	53.2	21.1	4.0	
Treated bagasse	36 - 123	124 - 231	232 - 357	358 - 439	440 - 600	96.6
	3.7	11.5	54.7	24.1	2.6	

sition step while enhances the fourth and fifth decomposition stages. This reflects that the presence of propionic acid strongly affected the structure of bagasse, *i.e.*, a good interaction between bagasse and propionic acid. So, the results of thermal analysis indicated that propionic acid should affect the physico-chemical properties of bagasse.

3.3. Effect of pH

The pH of the dye solution has a great influence on the adsorption of the dye on the biosorbent presumably due to the effect on the surface properties of the adsorbent and ionization or dissociation of the dye molecules. **Figure 3** represents the effect of pH on the removal of both dyes. The highest dye removal efficiency was observed at pH = 1.9 and pH = 2.1 for reactive yellow 2 and reactive blue 4, respectively.

These acidic pH values might correspond to the rate of dissociation of the sulphonic acid groups located on the dyes. Furthermore, it is assumed that under acidic conditions the hydroxyl groups of bagasse and the carboxylic group of propionic acid are protonated *i.e.*, $-\text{OH}_2^+$ and $-\text{COOH}_2^+$. Thus a significant electrostatic interaction may occur between the positively charged sites of the adsorbent and the negatively charged part of the two reactive dyes (sulphonate groups) [15]. The lower adsorption of both dyes at alkaline pH values may be due to the presence of excess OH^- ions competing with the dye anions for the adsorption sites.

3.4. Effect of Agitation Time and Initial Dye Concentration

The relation between removal of both dyes and the contact time was studied and the results are shown in **Figure 4**. It appears that removal of both dyes was rapid in the initial stages and then steadily increases with increasing time.

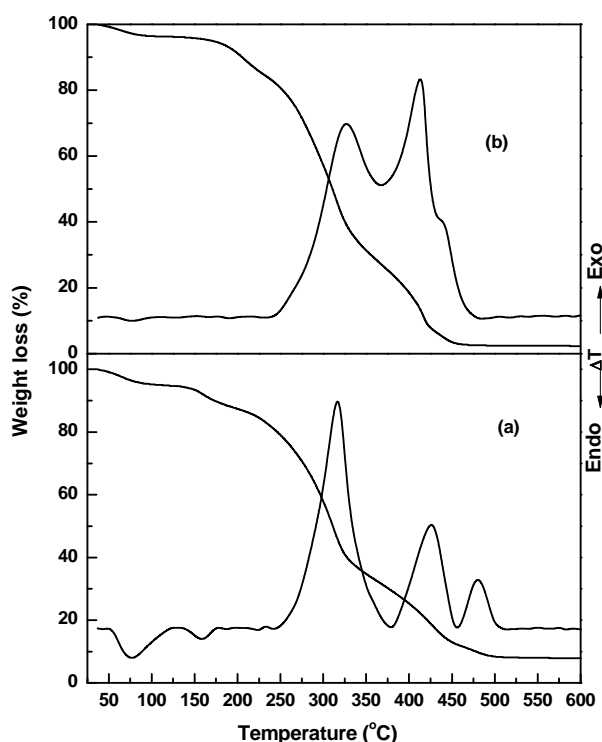


Figure 2. TGA and DTA curves of (a) untreated bagasse and (b) bagasse treated with 5 wt% propionic acid.

The rapid removal at the initial contact time may be ascribed to the availability of high positive charges on the bagasse surface for adsorption of the anionic reactive dyes. The subsequent slow adsorption of the two dyes is presumably correlated with the electrostatic repulsion between the adsorbed species onto the bagasse surface and the negative charge adsorbate species existing in the solution besides the slow pore diffusion of the dye into the bulk of the adsorbent [16]. **Figure 5** shows the influence of the initial concentration of both dye solutions on the

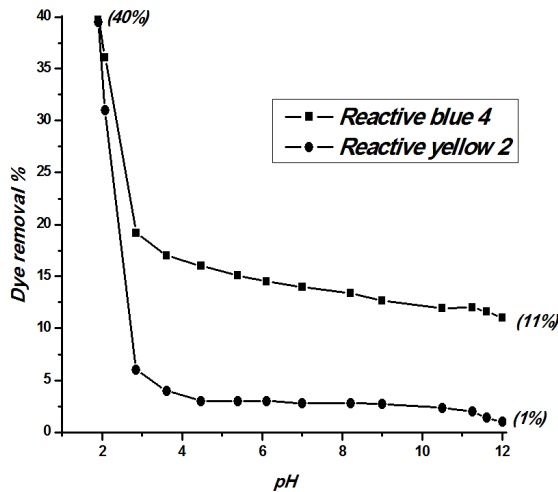


Figure 3. Effect of pH on dye removal, (1 g of 1 mm particle size).

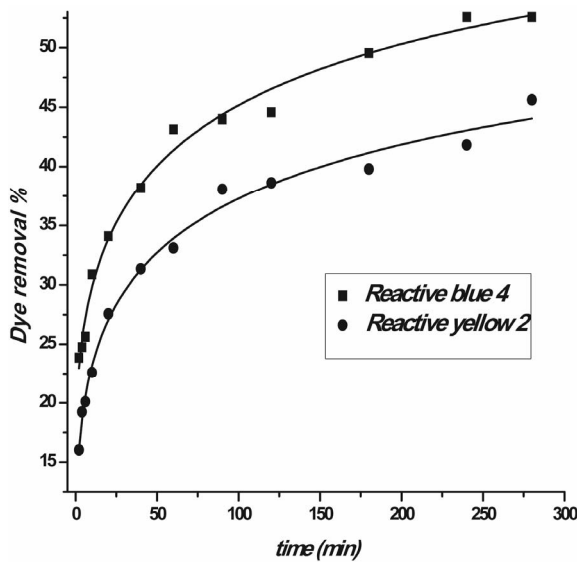


Figure 4. Effect of agitation time on dye removal.

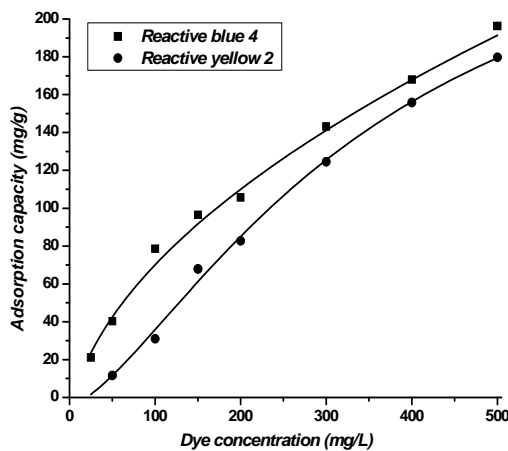


Figure 5. Effect of initial dye concentration on dye removal.

adsorption dynamics by bagasse treated with propionic acid. Upon raising the initial dye concentration of the reactive yellow 2 from 50 to 500 mg/L the adsorbent capacity increased from 12.5 to 180 mg/g whereas for reactive blue 4 the capacity increases from 20 to 200 mg/g when the initial dye concentration increased from 25 to 500 mg/L.

3.5. Effect of Sorbent Particle Size

The effect of particle size on the adsorption of the two dyes onto the modified bagasse was studied (Figure 6), using a fixed sorbent dose (25 mg/50 ml). Different particle sizes range i.e., 0.25, 0.5, 0.71 and 1 mm were adopted. The results reveal that the percentage removal of the dyes increases with the decrease in bagasse particle size. Clearly a decrease in particle size would increase the surface area and consequently an increase in dye adsorption onto the bagasse surface occurs. Various factors are responsible for the low adsorption capacity of the dyes on the sorbent large particles. Among them are the diffusional path length or mass transfer resistance, contact time and blockage sections of the particles (not utilized for adsorption) [17].

3.6. Effect of Sorbent Dosage

The effect of adsorbent mass on the adsorption of the two dyes was investigated on treated bagasse of 1 mm particle size at initial dye concentration of 500 mg/L. Figure 7 depicts the expected pattern for both dyes where the percent removal of each dye increases as the adsorbent dosage increases. This may be attributed to the increase in surface area of bagasse and availability of adsorption sites for the two reactive dyes. However, the adsorption density, the amount adsorbed by unit mass, decreases. This may be due to unsaturation of adsorption sites or

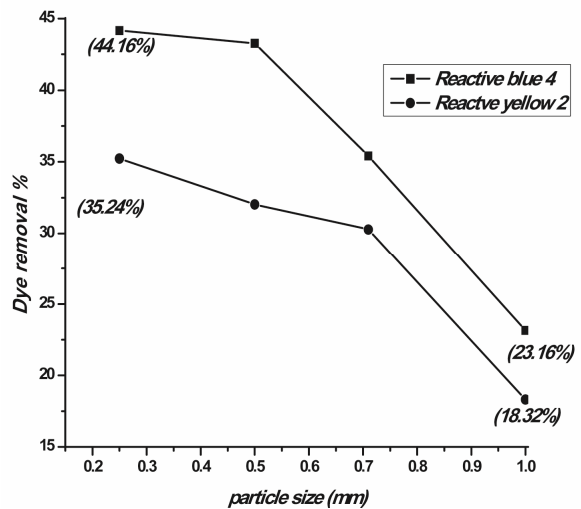


Figure 6. Effect of sorbent particle size on dye removal.

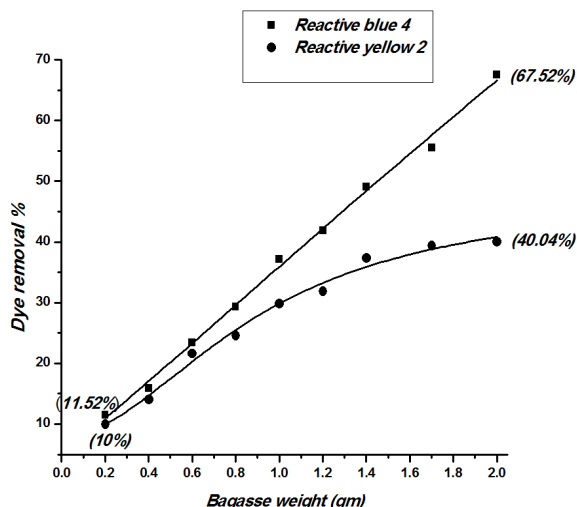


Figure 7. Effect of bagasse dosage on dye removal.

due to some sort of particle interaction such as aggregation which will decrease the total surface area of adsorbent and consequently an increase in the diffusion path length [18]. It is clear from the obtained results that the removal of reactive blue 4 is more pronounced relative to that of reactive yellow 2 upon increasing of the bagasse weight above 1 gm.

3.7. Adsorption Isotherms

The adsorption isotherms are important to optimize the design of an adsorption system to remove the dye. The parameters obtained from the different isotherms provide important information about the adsorption mechanisms and surface properties. The most widely accepted surface adsorption models are those of Freundlich and Langmuir.

3.7.1. Freundlich Isotherm

Freundlich isotherm is expressed as.

$$q_e = K_F C_e^{1/n} \quad (2)$$

where q_e (mg/g) is the amount of dye adsorbed at equilibrium, C_e (mg/L) is the equilibrium liquid-phase concentration of the dye and K_F is a constant related to the bonding energy and adsorption capacity. $1/n$ indicates the adsorption intensity of dye onto the adsorbent and the type of isotherm to be irreversible ($1/n = 0$), favorable ($0 < 1/n < 1$) and unfavorable ($1/n > 1$).

Equation (2) can be arranged to a linear form:

$$\log q_e = \log K_F + (1/n)\log C_e \quad (3)$$

To study the applicability of the Freundlich equation for dye absorption onto bagasse a linear plot of $\log q_e$ versus $\log C_e$ is plotted and presented in Figure 8 and the calculated parameters for Freundlich linear equation are shown in Table 3. The reactive yellow 2 adsorption by

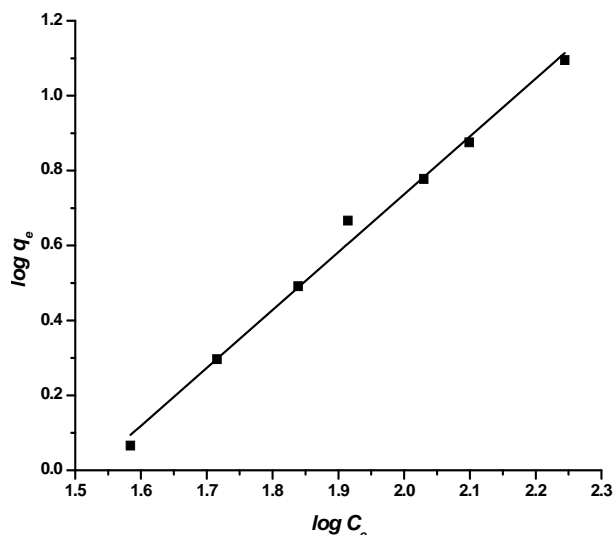


Figure 8. Freundlich isotherm for the sorption of reactive yellow 2 at pH 1.9.

Table 3. Langmuir and Freundlich isotherm parameters for reactive yellow 2 and reactive blue 4 on modified bagasse.

Dye	Freundlich		
	R ²	KF	1/n
Reactive yellow 2	0.9916	0.0045	1.5439
Reactive blue 4	-	-	-
Dye	Langmuir		
	R ²	Q _m (mg/g)	a(L/mg)
Reactive yellow 2	-	-	-
Reactive blue 4	0.9956	13.2	0.052

modified bagasse fitted the Freundlich model well with $R^2 = 0.9916$. The linear plot suggests the applicability of the Freundlich equation. The $1/n$ is lower than 1.0, indicating that reactive yellow 2 is favorably adsorbed by the pre-treated bagasse. On the other hand the linear plot of the Freundlich equation is not valid for reactive blue 4.

3.7.2. Langmuir Isotherm

The Langmuir equation is expressed as follows:

$$q_e = aQ_m C_e / (1 + aC_e) \quad (4)$$

where q_e and C_e are defined as in Freundlich equation, Q_m is the maximum adsorption capacity and a is the Langmuir constant. Its linear form is expressed as:

$$C_e/q_e = (1/a Q_m) + (C_e/Q_m) \quad (5)$$

The Q_m and a values can be obtained from the slope ($1/Q_m$) and the intercept ($1/a Q_m$) of the linear plot C_e/q_e versus C_e . Figure 8 illustrates the linear form of Langmuir isotherm of the reactive blue 4 and the calculated pa-

rameters are included in **Table 3**. It is clear from **Figure 9** that the adsorption of the reactive blue 4 fits very well the Langmuir equation with $R^2 = 0.9956$ while the data do not fit with the Freundlich equation .

3.8. Adsorption Kinetics

In order to investigate the adsorption process of the reactive dyes by pretreated bagasse, pseudo-first order (Equation (6)) and pseudo-second order (Equation (7)) kinetic models were used.

$$\log(q_e - q_t) = \log q_e - k_1 t / 2.303 \quad (6)$$

And

$$t/q_t = 1/h + t/q_e \quad (7)$$

where q_e is the amount of dye sorbed at equilibrium (mg/g), q_t is the amount of dye sorbed at time t (mg/g), k_1 is the rate constant of the pseudo-first order sorption (min^{-1}), h ($k_2 q_e^2$) is the initial adsorption rate (mg/g-min) and k_2 is the rate constant of pseudo-second order kinetics (mg/g min). **Figure 10** shows that the pseudo first order equation fits well for the adsorption of reactive yellow 2 whereas the pseudo-second order equation is not valid. Moreover, the correlation coefficient R^2 and K_1 were calculated from the intercept and slope respectively and presented in **Table 4**. The linear plot of t/q_t versus t for reactive blue 4 shows a good linear relationship, **Figure 11**. On the other hand the applicability of the pseudo-first order equation on the adsorption of reactive blue 4 was found not applicable. The correlation coefficient R^2 and K_2 were calculated from the intercept and the slope respectively and recorded in **Table 4**.

From **Table 4**, the correlation coefficients for the first and second order kinetic models are greater than 0.99 which led to believe that the first and second order equa-

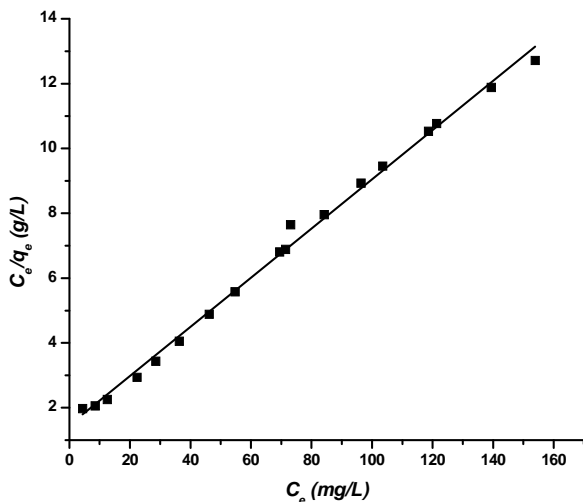


Figure 9. Linear form of the Langmuir isotherm for reactive blue 4 at pH 2.1.

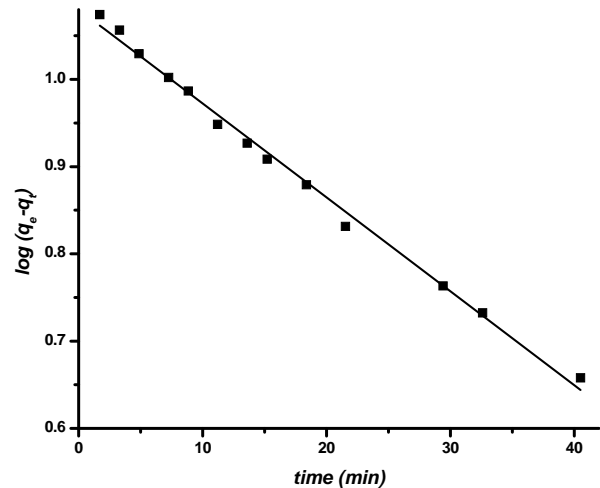


Figure 10. Pseudo-first order plot for the desorption of reactive yellow 2 at pH 1.9.

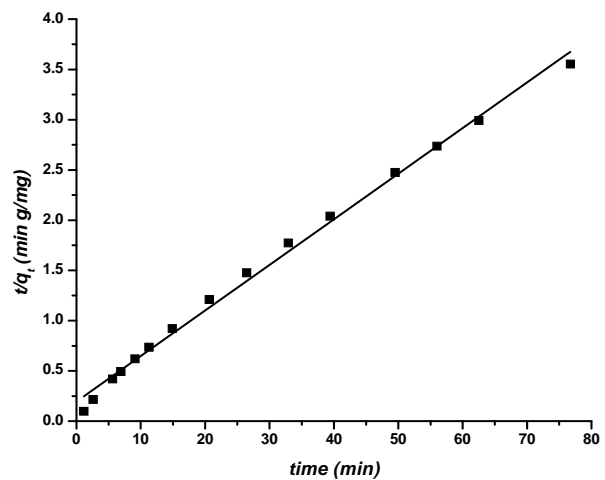


Figure 11. Pseudo-second order plot for adsorption of reactive blue 4 at pH 2.1.

Table 4. Comparison of the first and second order adsorption rate constants and the correlation coefficients.

Dye	Pseudo-first order		Pseudo-second order	
	R^2	K_1 (min^{-1})	R^2	K_2 ($\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1}$)
Reactive yellow 2	0.9945	0.0248	-	-
Reactive blue 4	-	-	0.9950	0.0106

tions provided good correlations for the biosorption of reactive yellow 2 and reactive blue 4 respectively, on modified bagasse.

3.9. Intra-Particle Diffusion

The intra-particle diffusion model was applied to test the

possibility of intra-particle diffusion of reactive yellow 2 and reactive blue 4 into pre-treated bagasse. The adsorption process involves a transport of solute from the aqueous phase to the surface of the solid particles with a subsequent diffusion of the dye molecules into the interior pores which is a slow process and is therefore, a rate determining step. The particle diffusion model is expressed by the following equation [19]:

$$q_t = K_d t^{1/2} + C \quad (8)$$

where C (mg/g) is the intercept and K_d is the intra-particle diffusion rate constant ($\text{mg/g}\cdot\text{min}^{-1/2}$). Two intersecting linear plots are obtained for both dyes indicating diffusion of the dye molecules into macro- and micropores of bagasse. If the mechanism of adsorption follows on intra-particle diffusion process, the plot of q_t versus $t^{1/2}$ should be straight line and pass through origin. **Figure 12** shows that there are two separate regions that represent the steps involved during the process of adsorption. The initial straight line is due to the bulk diffusion while the second straight line corresponds to the intra-particle diffusion for both dyes. The plots were not best-fit straight lines passing through the origin. This phenomenon indicates that there is a boundary layer resistance. One of the possible reasons for the deviation of the straight line from the origin is that there is a difference in the rate of mass transfer in the initial and final stages of dye adsorption. The results of the experiment suggest that the intra-particle diffusion is not the only step controlling adsorption of the two dyes [20].

3.10. Desorption and Reuse

A further goal of any adsorbent used is reuse potential. So, before the investigation of reuse, the adsorption experiments were carried out to find the optimum pH-desorption conditions. **Figure 13** shows the effect of pH values on the desorption process of the two dyes. It shows that the two dyes are stable at lower pHs whereas increasing pH higher than 10 results in a rapid desorption of both dyes from bagasse surface. It is thus to be concluded that the two reactive dyes are stable in the pH range 3 - 10 for reactive yellow 2 and 6 - 10 for reactive blue 4 while they may be completely recovered from the treated bagasse surface at pH = 12 and higher. It is of interest to mention here that these results are in agreement with the results presented in **Figure 2** which show that the remarkable dye removal of both dyes on modified bagasse was at lower pH values.

4. Conclusion

Modified bagasse by propionic acid is an effective biosorbent for the removal of both reactive yellow 2 and reactive blue 4 dyes from wastewater. The maximum

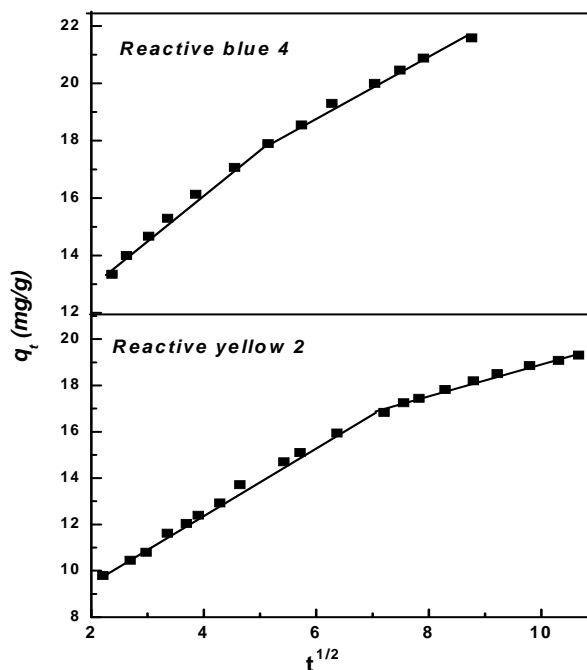


Figure 12. Intra-particle diffusion model for reactive yellow 2 at pH 1.9 and reactive blue 4 at pH 2.1.

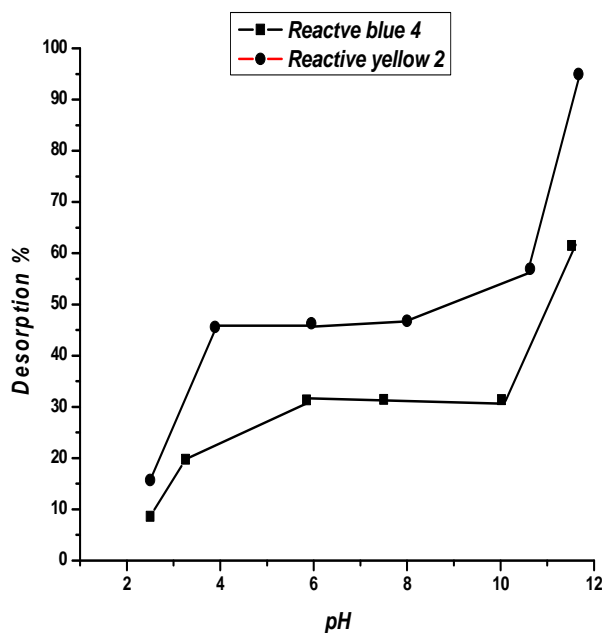


Figure 13. Effect of pH on the desorption process of the two dyes.

removal was observed at pH = 1.9 and pH = 2.1 for reactive yellow 2 and reactive blue 4, respectively. Moreover, the decrease of particle size of adsorbent was accompanied with a pronounced removal of both dyes. The equilibrium adsorption was best fitted with the Freundlich isotherm for reactive yellow 2 and with the Langmuir isotherm for reactive blue 4. Adsorption kinetics revealed

that reactive yellow 2 is well described by a pseudo-first order whereas reactive blue 4 fits good for pseudo-second order. Modified bagasse can be reused by desorption of both dyes in the pH = 12 and higher. The hydroxyl and carboxylic groups from bagasse and propionic acid respectively enhance the removal of both reactive dyes.

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