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### Application of Acid Treated Almond Peel for Removal and Recovery of Brilliant Green from Industrial Wastewater by Column Operation

Rais Ahmad<sup>a</sup>; Pijush Kanti Mondal<sup>a</sup>

<sup>a</sup> Environmental Research Laboratory, Department of Applied Chemistry, Faculty of Engineering and Technology, Aligarh Muslim University, Aligarh, UP, India

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## Application of Acid Treated Almond Peel for Removal and Recovery of Brilliant Green from Industrial Wastewater by Column Operation

Rais Ahmad and Pijush Kanti Mondal

Environmental Research Laboratory, Department of Applied Chemistry,  
Faculty of Engineering and Technology, Aligarh Muslim University,  
Aligarh, UP, India

**Abstract:** The treated almond peels (TAP) have been employed as adsorbents for the removal of Brilliant green dye from waste water. The nature of possible adsorbent and dye interaction was examined by the FTIR and SEM technique. The adsorption of BG was found to be maximum (93%) at pH 8. The extent of removal of BG was found to be dependent on the adsorbent dose, the temperatures, and the times. The equilibrium data for adsorption was best represented by the Langmuir isotherm. Thermodynamic parameters ( $\Delta H^0$  and  $\Delta G^0$ ) suggest endothermic and spontaneous process. Kinetic studies show better applicability of second-order kinetic model. The practical utility of TAP was demonstrated by removing BG from pure solution and industrial effluent system by column process. It was found that the removal efficiency of TAP was better by pure solution. The breakthrough capacities of pure solution and industrial effluent systems on TAP are found to be 30 and 17 mg/g, respectively.

**Keywords:** Biosorption, breakthrough, capacity, characterization, desorption, isotherm, kinetics

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Address correspondence to Rais Ahmad, Environmental Research Laboratory, Department of Applied Chemistry, Faculty of Engineering and Technology, Aligarh Muslim University, Aligarh, UP, 202 002, India. Tel.: +919411491160. E-mail: raisdye@gmail.com

## INTRODUCTION

The importance of dyes to human civilization both ancient and contemporary is well documented. Environment compliance requirements have become increasingly difficult to both wastewater discharge and chemical handling (1). Discharge of colored wastewaters from various industries such as textile dyeing, paper and pulp, and food processing industries is currently a major problem for environmental management especially in developing countries. Approximately 10,000 different dyes and pigments are used industrially and over  $7 \times 10^5$  tons of tons of these dyes are produced annually worldwide (2). Dyes in water affect the nature of the water, inhibiting sunlight penetration into the stream, and reducing the photosynthesis reaction. Some dyes are also toxic and even carcinogenic (3,4). The conventional methods for treating dye-containing wastewaters include coagulation and flocculation, reverse osmosis, and precipitation (5,6). However, these technologies do not exhibit significant effectiveness and also economically nonviable for the removal of dyes. Adsorption of dyes onto solid/water interface has been found to be an efficient and economically cheap process (7) and an effective method to control the extent of water pollution due to dyes. The most commonly used adsorbent for the dye removal is activated carbon (8) but it is relatively expensive due to its high cost of regeneration. The use of some low cost adsorbents are available in the literature for dye removal from wastewater which included apple pomace, wheat straw (9), orange peel (10), banana peel (11), maize cob (12), maize stalk (13), rice husk (14), barley husk (15), peanut hull (16), wood chip (17), palm fruit bunch (18,19), saw dust (20), bark (21), leaf (22), coil pith (23), banana peel (24), bagasse pith (25), and aquatic plants (26,27). However, there is further need to search easily available, low-cost material, which have no economical use and therefore can be utilized for the removal of dyes.

The present study is concerned with experimental investigation on treated almond peel (TAP), which is a low-cost material having the percent adsorption (93) while raw almond peels shows 86% and abundantly available in the northern region of India. The material can also be used to remove and recycle the industrial waste from the carpet industry. This material has better adsorption capacity [ $q_m = 123.41 \text{ mg/gm}$ ] as compared to other materials such as orange peel [ $q_m = 20.5 \text{ mg/gm}$ ] (10), rice husk [ $q_m = 86.9 \text{ mg/gm}$ ] (14), banana peel [ $q_m = 21 \text{ mg/gm}$ ] (24), and wheat straw [ $q_m = 19.82 \text{ mg/gm}$ ] (9). The effects of various parameters such as contact time, adsorbent dose, temperature, and pH on adsorption have been studied.

## MATERIALS AND METHODS

### Preparation of Adsorbent Materials

The naturally dried almond peels (AP) were procured locally. The adsorbent was sieved through 50–100 (BSS) mesh size. Acid treatment on the almond peel was performed by immersing 10 g of the original almond peel in 100 ml of 10% hydrochloric acid (HCl) and left for 4 h. The sample was then filtered using Whatman No.1 filter paper to separate the almond peel from the solution. The almond peel was then rinsed with deionized water and dried in an oven overnight at 105°C. The chemically treated almond peel was stored in desiccators to prevent moisture build up and used as such.

### Adsorbate Solution

Brilliant green (BG) supplied from CDH (India) were used as obtained. All other chemicals used in this study were reagent grade in DDW. The industrial effluent having [chemical oxygen demand (COD) around 2200 mg/L, volatile suspended solid (VSS) content of 2.5 g/L] was obtained from secondary clarifier of the Carpet industry, Moradabad, India.

## ADSORPTION STUDIES

Adsorption studies were carried out by batch and column process. In the batch process 0.5 g adsorbent was placed in a conical flask in which 50 ml solution of BG of desired concentration was added and the mixture was shaken in temperature controlled shaker incubator for 3 h at 120 rev./min. The mixture was then filtered using Whatman filter paper number 41 and final concentration of dye was determined in the filtrate by UV-visible spectrophotometer (Elico, SI-164). In the column process 0.5 g of adsorbent was placed in a glass column (0.6 cm internal diameter) with glass wool support. BG solution was passed through the column with a flow rate of 1 ml/min. The effluent was collected in fractions and the amount of BG in each fraction was determined by UV-VIS. The amount of dye adsorbed was calculated by subtracting final concentration from initial concentration.

## BREAKTHROUGH CAPACITY AND DESORPTION STUDIES

0.5 g of adsorbent was taken in a glass column (0.6 cm internal diameter) with glass wool support. One thousand milliliters of dye solution with

20 mg/l initial concentration ( $C_0$ ) was passed through the column with a flow rate of 1 ml/min. The effluent was collected in 50 ml fractions and the amount of dye (C) was determined in each fraction by UV-VIS spectrophotometer. The breakthrough curve of dye and industrial effluent (after digestion) were obtained by plotting  $C/C_0$  versus volume of the effluent. In order to see the desorption behavior, the exhausted column was washed several times with double distilled water to remove excess of dye from the column, then 0.5 M acetic acid was passed separately through each column containing adsorbed BG and effluent. The BG eluted was collected in 10 ml fractions. The flow rate was maintained at 1 ml/min in each case.

## RESULTS AND DISCUSSION

### Characterization of the Adsorbent

The FTIR spectra of TAP in the range of (400–4000)  $\text{cm}^{-1}$  were taken to obtain information on the nature of the possible adsorbent-dye interactions and are presented in (Fig. 1). The FTIR spectra of the TAP displayed the following bands 3572  $\text{cm}^{-1}$  (O–H stretching vibrations in carboxylic acid), 2095  $\text{cm}^{-1}$  (C≡C stretching vibrations), 1559  $\text{cm}^{-1}$  (C=C stretching vibration in aromatic rings), and 1086  $\text{cm}^{-1}$  (C–OH stretching vibrations). The FTIR spectra obtained was in agreement with the results reported in the studies (28), which reported that a more acidic group such as carboxylic was produced by acid treatment of the adsorbent.

Scanning electron micrograph (SEM) was recorded at two different magnifications Figs. 2a, 2b using a software controlled digital scanning electron microscope–LEO 420. The SEM clearly reveal the surface texture and porosity of almond peels (AP) with holes and a small opening

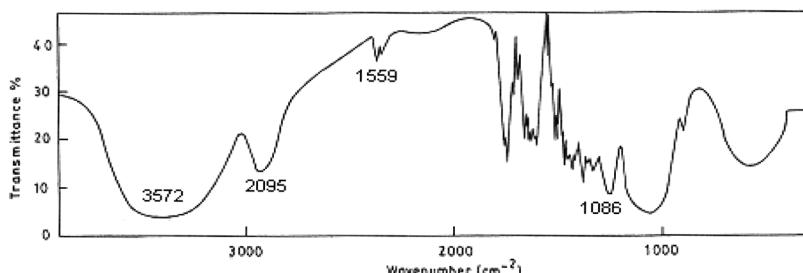
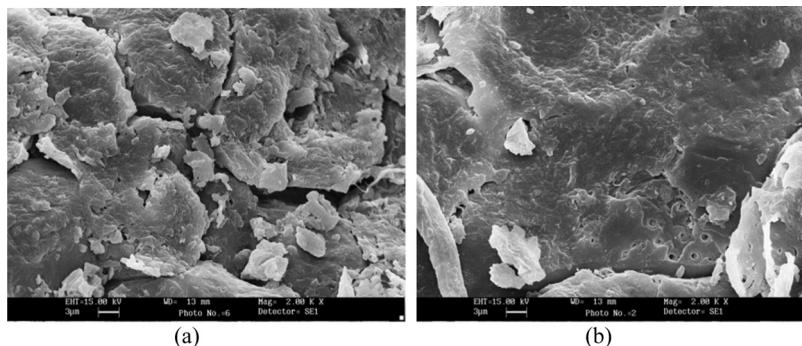


Figure 1. FTIR spectra of treated Almond peel.



**Figure 2.** Scanning electron micrographs of treated Almond peel before (a) and after (b) adsorption.

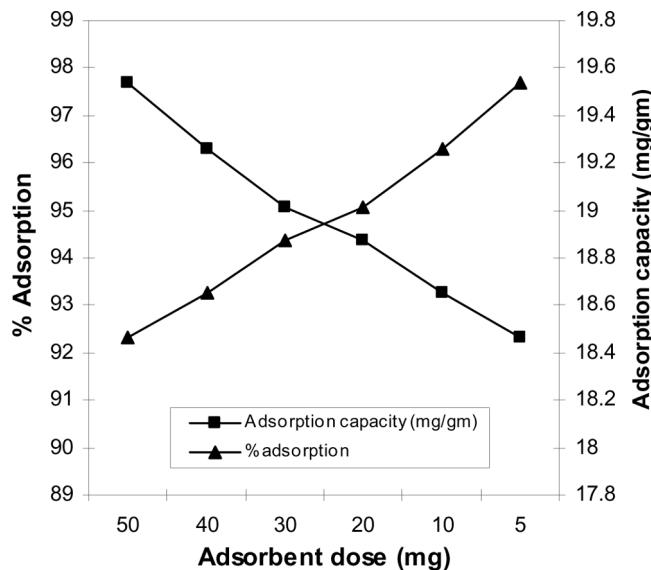
on the surface before adsorption while the SEM study after adsorption clearly indicates that these small opening and pores are filled probably because of the adsorption of brilliant green dye.

### Effect of Adsorbent Doses

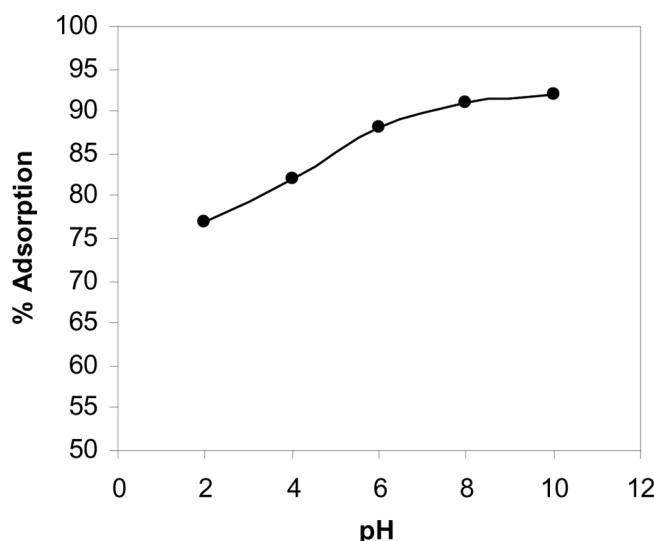
The adsorption capacity (mg/g) and percentage (%) adsorption of BG by varying the dose of TAP at 50°C temperature is shown in Fig. 3. The % adsorption increases as the adsorbent dose of TAP is increased, while adsorption density/capacity decreases. The decrease in adsorption density may be due to the fact that some adsorption sites may remain unsaturated during the adsorption process whereas the number of sites available for adsorption increases by increasing the adsorbent doses and that results in the increase in removal efficiency.

### Effect of pH

Figure. 4 shows the effect of pH on the removal of BG on the treated almond peels from aqueous solution. It was observed that the BG removal was highly dependent on the pH of the solution which affected the surface charge of the TAP and the degree of ionization of the adsorbate. It was found that an increase in the solution pH led to an increase in the BG removal efficiency. The percent removal of BG increased from 77% to 92% by varying the solution pH from 2 to 10. At lower pH, more protons were available (excess H<sup>+</sup> ions), thereby decreasing the electrostatic attractions between positively charged dye anions and positively



**Figure 3.** Effect of adsorption dose on % adsorption and adsorption capacity of BG on TAP at 50°C.



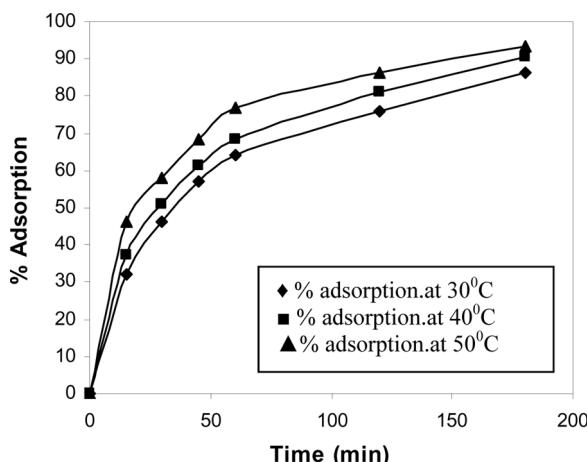
**Figure 4.** Effect of pH.

charged adsorption sites and therefore the ionic repulsion between the positively charged surface and the cationic dye molecules caused a decrease in BG adsorption. The adsorption of BG was found to be maximum (93%) at pH 8, so all the experiment was done at pH 8.

### Effect of Temperature and Thermodynamic Parameters

The effect of temperature on adsorption of BG on the TAP was investigated by varying the adsorption temperature at 30, 40 and 50°C. Figure 5 shows the plot of % adsorption versus contact time at the three different temperatures. Referring to Fig. 5, the adsorption of BG on the TAP was found to gradually increase when the temperature was increased from 30 to 50°C. Increasing the temperature is known to increase the rate of diffusion of the adsorbate molecules across the external boundary layer and in the internal pores of the adsorbent particle, owing to the decrease in the viscosity of the solution (28). The enhancement in the adsorption capacity might be due to the chemical interaction between the adsorbate and the adsorbent. It was also noted that the % adsorption increased with increasing in temperature. It might be due to the possibility of an increase in the porosity and in the total pore volume of the adsorbent, an increase of the number of active sites for the adsorption, as well as an increase in the mobility of the BG molecules.

The thermodynamic parameters that must be considered to determine the process are changes in standard enthalpy ( $\Delta H^0$ ), standard



**Figure 5.** Effect of contact time with different temperature.

entropy ( $\Delta S^0$ ), and standard free energy ( $\Delta G^0$ ) due to transfer of the unit mole of the solute from the solution onto the solid-liquid interface. The value of  $\Delta H^0$  and  $\Delta S^0$  were computed using the following equation: The value of  $\Delta H^0$  and  $\Delta S^0$  were computed using the following equation:

$$\ln K_d = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (1)$$

Where  $R$  (8.314 J/mol K) is the universal gas constant,  $T$  (K) is the absolute solution temperature, and  $K_d$  is the distribution coefficient which can be calculated as:

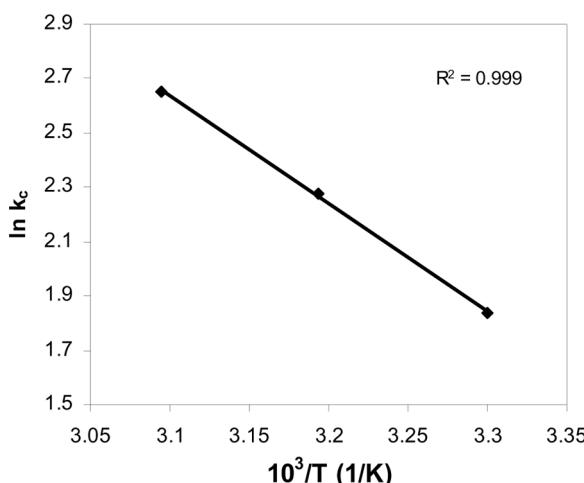
$$K_d = \frac{C_{Ae}}{C_e} \quad (2)$$

Where  $C_{Ae}$  (mg/l) is the amount adsorbed on solid at equilibrium and  $C_e$  (mg/l) is the equilibrium concentration.

The values of  $\Delta H^0$  and  $\Delta S^0$  were calculated from the slope and intercept of van't Hoff plots of  $\ln K_d$  versus  $1/T$  (Fig. 6).  $\Delta G^0$  can be calculated using the relation below:

$$\Delta G^0 = -RT \ln K_d \quad (3)$$

The calculated values of  $\Delta H^0$ ,  $\Delta S^0$  and  $\Delta G^0$  are listed in Table 1. The positive value of  $\Delta H^0$  indicated the endothermic nature of the adsorption interaction. The positive value of  $\Delta S^0$  showed the increased disorder at the solid/solution interface during the adsorption of BG on the TAP.



**Figure 6.** Van't Hoff plot for the adsorption.

**Table 1.** Thermodynamic parameters for adsorption

Temp. (°C)	ΔG <sup>0</sup> (KJ mol <sup>-1</sup> )	ΔH <sup>0</sup> (KJ mol <sup>-1</sup> )	ΔS <sup>0</sup> (KJ mol <sup>-1</sup> )
30	-50.33		
40	-60.49	33.053	
50	-68.18		0.124

The positive values of  $\Delta S^0$  reflect the affinity of TAP for BG dye and also it shows the increased randomness at the solid/solution interface. The adsorption increased randomness at the solid/solution interface with some structural changes in the adsorbate and adsorbent and an affinity of the cript adsorbent. The negative value of  $\Delta G^0$  indicated the feasibility of the process and the spontaneous nature of the adsorption with a high preference of BG onto the TAP.

### Adsorption Isotherms

The langmuir isotherm (29) is based on an assumption that the adsorption occurs at specific homogeneous sites within the adsorbent and the monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform strategies of adsorption with no transmigration of the adsorbate in the plane of the surface. The linear form of the Langmuir isotherm equation is given as:

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{1}{q_m} C_e \quad (4)$$

Where  $C_e$  is the equilibrium concentration of adsorbate (mg/l);  $q_e$  the adsorption capacity (mg/g);  $b$  and  $q_m$  are the Langmuir constants. The values of the Langmuir constants  $b$  and  $q_m$  were calculated from the slope and intercept of the linear plot of  $C_e/q_e$  versus  $C_e$  (Fig. 7). The coefficient  $b$  in the Langmuir equation is a measure of the stability of the complex formed between the dye and the adsorbent under specified experimental conditions.

The essential feature of the Langmuir isotherm can be expressed in terms of dimensionless constant separation or equilibrium parameter ( $R_L$ ), which is defined as:

$$R_L = \frac{1}{1 + bC_0} \quad (5)$$

Where  $C_0$  is the initial dye concentration (mg/l) and  $b$  is the Langmuir constant. The value of  $R_L$  calculated from different initial

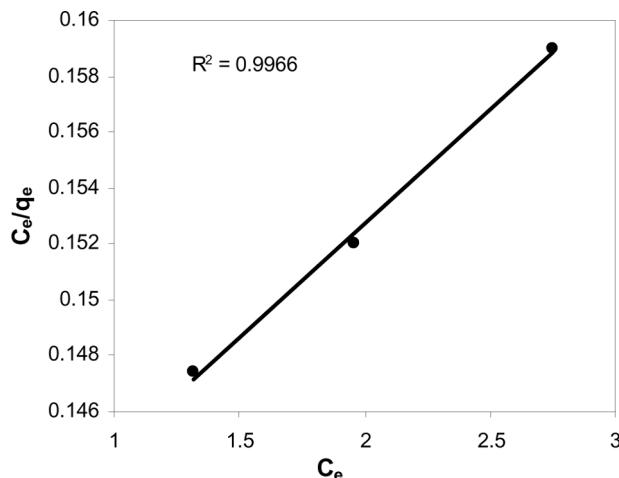


Figure 7. Langmuir plot for the adsorption of BG on TAP.

concentrations is reported in Table 2. The value of  $R_L$  indicates the type of the isotherm to be either unfavourable ( $R_L > 1$ ), linear ( $R_L = 1$ ), favorable ( $0 < R_L < 1$ ), or irreversible ( $R_L = 0$ ) (30). The  $R_L$  for BG is 0.9015. The values are in between 0 and 1 showing favorable adsorption on TAP.

The freundlich isotherm on the other hand assumes heterogeneous surface energies, in which the energy term in the Langmuir equation varies as a function of the surface coverage (30). The well-known linear form of the Freundlich isotherm (31) is given by the following equation:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (6)$$

Where  $C_e$  is the equilibrium concentration of the adsorbate (mg/l),  $q_e$  is the amount of adsorbate adsorbed per unit mass of adsorbent (mg/g),  $K_F$  and  $n$  are Freundlich constants with  $n$  giving an indication of how

Table 2. Langmuir and Freundlich constant for the adsorption of BG

Dye	Langmuir constant			Freundlich constant			
	$q_m$ (mg/g)	$b$ (l/mg)	$R^2$	$R_L$	$k_f$ (mg/g)	$n$	$R^2$
BG	123.41	0.001	0.99	0.98	19.14	0.64	0.96

favorable the adsorption process.  $K_F$  (mg/g) is the adsorption capacity of the adsorbent which can be defined as the adsorption or distribution coefficient and represents the quantity of dye adsorbed onto TAP for a unit equilibrium concentration. The slope of  $1/n$  ranging between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero (32). A value for  $1/n$  below one indicates a normal Langmuir isotherms, while  $1/n$  above one is indicative of cooperative adsorption (33). The plot of  $\log q_e$  versus  $\log C_e$  (Fig. 8) gave a straight line with slope of  $1/n$  whereas  $K_F$  was calculated from the intercept value.

Table 2 summarizes all the constants, correlation coefficients, and  $R^2$  values obtained for the two isotherm models for adsorption of BG on the TAP. The Langmuir model yielded the best fit data, as the  $R^2$  values were relatively high close to unity.

### Adsorption Kinetics

Kinetics of adsorption is quite significant as it decides the residence time of the adsorbate at solid/solution interface and helps in determining the rate of the adsorption process. The adsorption of dyes from the liquid to solid phase can be considered as a reversible reaction with equilibrium established between the two phases. The kinetics of adsorption of BG on the TAP was studied by applying two different kinetic models. The

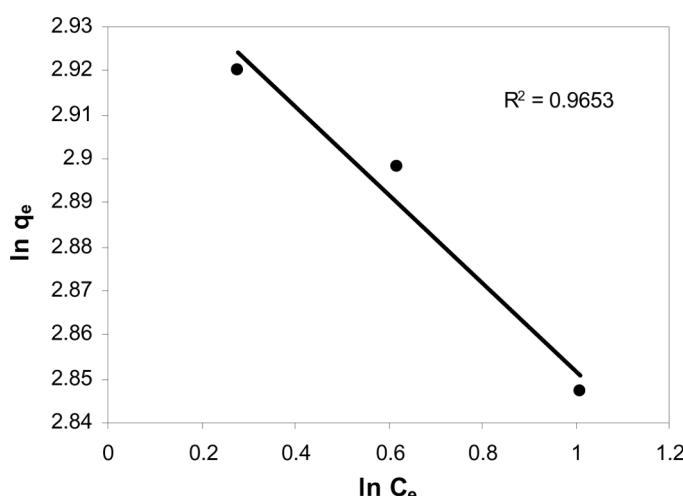


Figure 8. Freundlich plot for the adsorption of BG on TAP.

linear form of the pseudo first-order equation given by Lagergren and Svenska (34) is shown by Eq. (7).

$$\ln(q_e - q_t) = \ln q_e - K_1 t \quad (7)$$

where  $q_e$  and  $q_t$  are the amounts of BG adsorbed (mg/g) at equilibrium and at time  $t$  (h), respectively and  $k_1$  is the rate constant adsorption (1/h). Values of  $k_1$  were obtained from the slopes of the linear plots of  $\ln(q_e - q_t)$  versus  $t$ , as shown in Fig. 9.

On the other hand, the linear form of the pseudo-second-order equation (35) based on equilibrium adsorption is expressed as:

$$\frac{t}{q^t} = \frac{1}{K_2 q^2 e} + \frac{1}{q_e} t \quad (8)$$

Where  $k_2$  (g/mg h) is the rate constant of the second-order adsorption. If the second-order kinetic is applicable, the plot of  $t/q_t$  versus  $t$  should show a linear relationship, as shown in Fig. 10. Values of  $q_e$  and  $k_2$  can then be determined from the slope and the intercept of the plot. This procedure is more likely to predict the behavior over the whole range of adsorption.

Table 3 summarizes the rate constants and correlation coefficients,  $R^2$  of the two kinetic models for this adsorption process. The correlation coefficients,  $R^2$  values obtained from the two kinetic models. The second

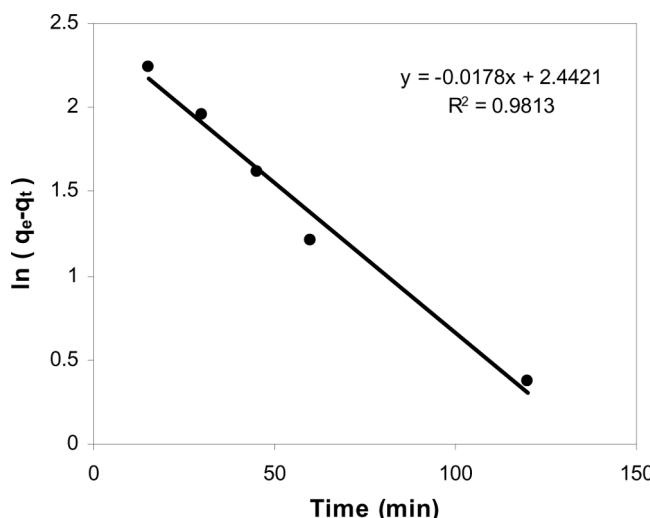
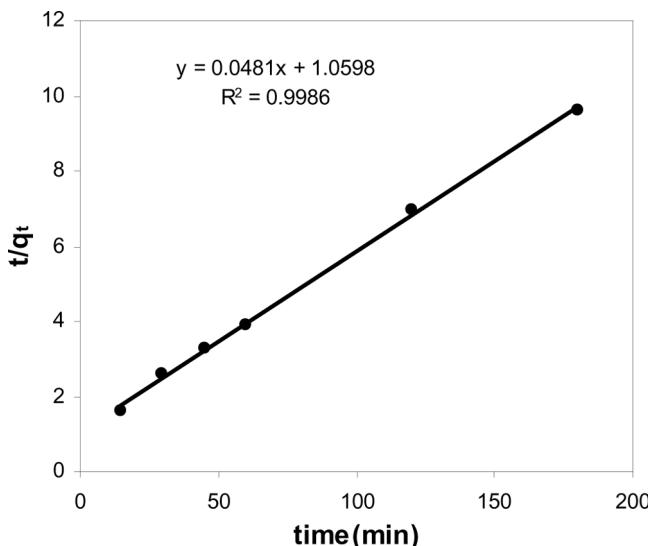


Figure 9. Representation of the kinetic data by pseudo first order model.



**Figure 10.** Representation of the kinetic data by pseudo second order model.

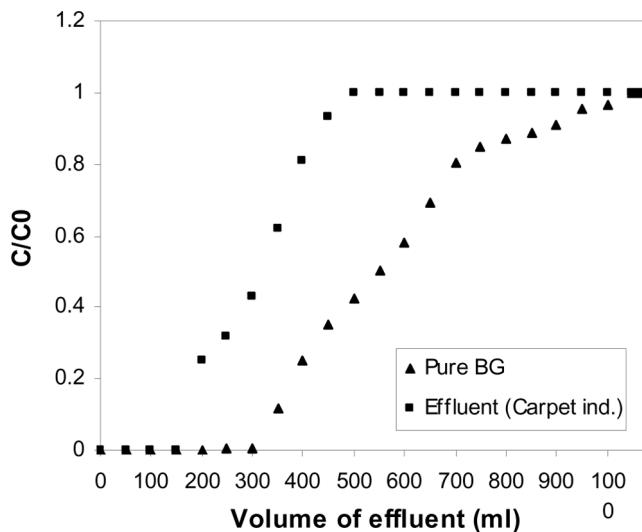
order-kinetic model yielded the best fit data, as the  $R^2$  values were relatively high (0.998) close to unity.

### Breakthrough Capacity

The breakthrough capacity of the sorbent is higher in case of the column process than the batch process. This is due to continuously large concentration at the interface of the sorption zone as the sorbate passes through the column while the concentration gradient decreases with time in a batch process. The breakthrough curve plotted in Fig. 11 reveals that pure brilliant green (300 ml) and industrial effluent (170) solution can be passed through the column of the adsorbent without any trace being detected in the effluent. The breakthrough capacity of the pure BG system and effluent system were calculated as 30 mg/g

**Table 3.** Pseudo first-order and pseudo second-order kinetic constants for the adsorption of BG

Pseudo first-order kinetic			Pseudo second-order kinetic		
$K_1$ (1/min)	$q_e$ (mg/g)	$R^2$	$K_2$ (g/mg min)	$q_e$ (mg/g)	$R^2$
0.0178	11.47	0.9813	0.00218	20.79	0.998

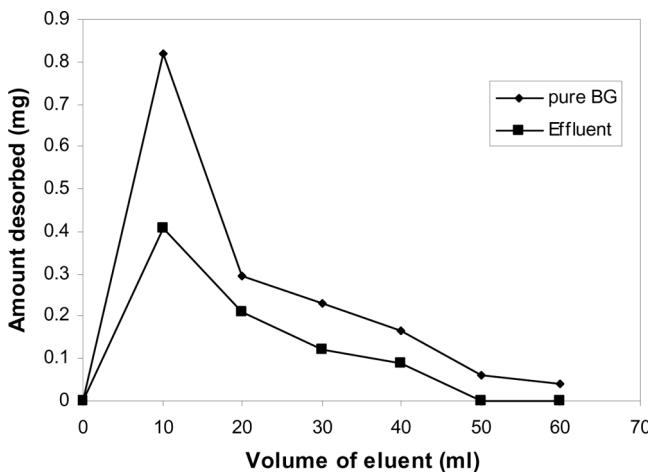


**Figure 11.** Breakthrough capacity curve for the adsorption of pure BG and Effluent on TAP.

and 17 mg/g. In order to explore the practical utility of the adsorbent, removal of BG was carried out by column processes using the pure BG system and effluent system (Carpet-industry) since, wastewater contains other organic and inorganic compounds. It is found that in pure BG adsorption system by column process is higher than the effluent system. The removal of BG by column process is light affected when the effluent system is used, showing that the removal efficiency of these dyes were different.

### Desorption Studies

Desorption studies help to elucidate the nature of adsorption recycling of the spent adsorbent and the dye. If the adsorbed dye can be desorbed using neutral pH water, then the attachment of the dye on the adsorbent is by weak bonds. If sulphuric acid or alkaline water can desorbs the dye, then the adsorption is by ion exchange. If an organic acid, like acetic acid can desorbs the dye, then the dye is held by the adsorbent through chemisorptions. Sodium hydroxide and sulphuric acid (1 M) did not show any desorption but weak acid, like acetic acid (0.5 M) solubilized about 65.23% of pure BG from the spent adsorbent (Fig. 12). Desorption of dye in acetic acid indicates that brilliant green dye is adsorbed onto AP through chemisorptions mechanism and very



**Figure 12.** Desorption studies of BG and Effluent adsorbed column by using 0.5 M Acetic acid as an eluent.

little desorption of dye in acid or base confirms the strong affinity of dye on the almond peel.

However, the recovery of dye is poor by the industrial effluent system (Fig. 12) as compared to the pure BG system. The % desorption of dye is slightly decreased in the effluent system. It is important to note that maximum elution of all the dye occur with in 10 ml fraction of the effluent, showing that the dye can be pre-concentrated effectively using TAP as adsorbent.

## CONCLUSION

1. The present investigation showed that HCl treatment could enhance the adsorption capacity of almond peel in removing BG from aqueous solutions and industrial effluent.
2. The adsorption studies indicate that the Langmuir model is better obeyed.
3. Thermodynamic parameters ( $\Delta H^0$  and  $\Delta G^0$ ) suggest that the adsorption process is endothermic and spontaneous.
4. The kinetics data are best fitted in the second-order rate equation as evident from the value of regression coefficients ( $R^2$ ).
5. The breakthrough capacities for pure BG solution and industrial effluent are found to be 30 and 17 mg/g, respectively. It has been found that the removal and recovery of BG is better by the pure as compared to the industrial effluent system.

6. The maximum efficiency of TAP has been demonstrated by eluting BG from exhausted columns and it has been found that 65% BG could be recovered while recovery of the effluent is 34% when 0.5 M acetic acid was used as eluent.

Therefore, TAP is an effective biosorbent and can be utilized for the removal and recovery of BG from wastewater. The process is also very cheap and economical and can be harnessed for the treatment of industrial waste on a large scale.

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