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# CEED-2

# Removal Of A Basic And Azo Dye From Aqueous Solution By Adsorption Using Activated Carbon

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# Abstract

The removal of dyes from colored effluents, particularly from textile industries, is one of the major environmental concerns these days. Current methods for removing dyes from wastewaters are costly and cannot effectively be used to treat wide range of such wastewaters. This work describes the use of commercial available activated carbon as an efficient adsorbent material for dye removal. Aqueous solutions of various basic dye Methylene Blue (MB) and azo-dye Tartrazine (TZ) concentrations 5-20 mg l<sup>-1</sup> and 10-100 mg l<sup>-1</sup>, respectively, were shaken with certain amount of adsorbents to determine the adsorption capacity. The effects of adsorbents dose, initial pH, initial dye concentration, agitation speed and contact time on dye removal have been studied. Maximum dye was removed from the solution within 60-90 min after the beginning of every experiment. Adsorption parameters were found to fit well into Langmuir and Freundlich adsorption isotherm models with correlation coefficient (R<sup>2</sup> > 0.99) in the concentration range of MB and TZ studied.

Keywords: Activated carbon, Adsorption, Methylene Blue, Tartrazine.

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# 1. Introduction

A large number of synthetic organic compounds (SOCs) have been produced for the purpose of industrial and domestic uses for many years. Most of them have been found to be toxic, carcinogenic and mutagenic. These compounds may enter the aquatic environment from atmospheric sources, industrial and municipal effluent, and agricultural runoff [1].

The textile industry plays a part in the economy of several countries around the world. Dyeing is a fundamental operation during textile fiber processing. This operation causes the production of more or less colored wastewater, depending on the degree of fixation of the dyestuffs on the substrates, which varies with the nature of the substances, the desired intensity of coloration, and the application method<sup>[2]</sup>. Effluents discharged from textile and dyeing industries are of low BOD and high COD. Disposal of this colored water into receiving water can be toxic to aquatic life. The dyes upset the biological activity in water bodies. They also pose a problem because they may be mutagenic and carcinogenic and can cause severe damage to human beings, such as dysfunction of kidney, reproductive system, liver, brain and central nervous system[3, 4]. The dye bearing effluents are considered to be very complex and inconsistent mixtures of many pollution substances ranging from organicchlorine based pesticides to heavy metals and are considered to be recalcitrant, nonbiodegradable and persistent[5]. Treatment of dye-based effluents is considered to be most challenging in the environmental fraternity and the industries; the recent stringent regulations complicate the problem further. Various treatment methods including, physical, physicochemical and chemical processes have been investigated for treating dye-bearing effluents [6-9]. All these methods have different color removal capabilities, capital costs and operating rates. Among these processes, adsorption has been found to be superior to other techniques for wastewater treatment in terms of initial cost, simplicity of design, ease of operation and insensitivity of toxic substances[10]. Activated carbon is the most widely used adsorbent with great success because of its high adsorption capacity[11].

Activated carbons are unique and versatile adsorbents because of their large surface area, microporous structure, high adsorption capacity, and high degree of surface reactivity. Their important applications are related to their use in removal of color, odor, taste, and other undesirable organic impurities from potable waters, in the treatment of domestic and industrial wastewater, solvent recovery, air purification in inhabited spaces such as restaurants, food processing and chemical industries, in air pollution control, and in a variety of gas phase applications[12].

The present study was undertaken to evaluate and compare the efficiency of the activated carbon for the removal of basic dye (Methylene Blue, MB) and azo-dye (Tartrazine, TZ) from aqueous solution. The removal efficiency was determined to study the effects of contact time, initial concentration of dye solution and pH, adsorbent dosage and agitation rate,

## 2. Experimental

# 2.1. Materials and Apparatus

The dyes used in this study were methylene blue dye (molecular formula:  $C_{16}H_{18}N_3ClS$ ) and Tartrazine (molecular formula:  $C_{16}H_9N_4Na_3O_9S_2$ ) supplied by Morgan Chemical Company and were used without further purification. Other reagents include dilute HCl and NaOH solutions. All reagents were of analytical grade. Deionized water was used throughout the experiment.The dyes concentrations were determined using Agilant UV-Vis Cary 60 PC scan double beam recording spectrophotometer using (1) cm glass cells. A digital pH meter, type 720 WTW 82362 was used to adjust the pH.

### 2.2. Preparation of adsorbent

Commercial available activated carbon (500  $\mu$ m  $\leq$  D  $\leq$  2mm) supplied by Morgan Chemical Company) was used as the adsorbent without further chemical treatment. A measured quantity was immersed in hot water for three hours. The washed activated carbon was filtered and oven dried at 60°C for 2 hours and then heated to 200 °C for 3 hours with flow of N<sub>2</sub>.

### 2.3. Physical characterization of activated carbon

The properties of the activated carbon adsorbent including particle size, density, pore volume and porosity were determined according to the method of American Standard for Testing & Materials (ASTM). The apparent surface area was measured from N<sub>2</sub> adsorption at 77K in a Quantrachrome Autosorb I-CLP. Total surface areas were calculated using the BET equation[11]. The values for density, total pore volume and BET surface area are 0.447 gcm<sup>-3</sup>, 0.124 cm<sup>3</sup>g<sup>-1</sup> and 776 m<sup>2</sup>g<sup>-1</sup> respectively.

### 2.4. Preparation of dye solution

A stock solution of the dye was prepared by dissolving 1.0 g of dye in 1000 ml distilled water to make a stock solution of 1000 mg l<sup>-1</sup>. The experimental solution was prepared by diluting definite volume of the stock solution to get the desired concentration. For absorbance measurements a spectrophotometer UV-VIS double beam PC scanning (Agilent. INC) was employed, the activated carbon was filtered before sampling. The maximum wavelength  $\lambda_{max}$  for the methylene blue was measured at 664 nm, while for tartrazine it was at 425 nm. Concentrations were determined from a standard calibration curve.

### 2.5. Adsorption Procedure

The adsorption isotherms have been determined by mixing of dyes solution (100 ml) of known initial concentration of MB (2-20 ppm) and TZ(10-100 ppm), and varied amounts of adsorbents (0.2-1.5 gm) were placed into volumetric flasks. The adsorption behaviors of the samples were studied by evaluating the percentage removal efficiency of methylene blue and Tartrazine calculated as:

Removal efficiency =  $[(C_0 - C)/C_0] \times 100$  (1) Where C<sub>0</sub> is the initial concentration of methylene blue, C is the solution concentration after

Where  $C_0$  is the initial concentration of methylene blue, C is the solution concentration after adsorption at any time.

The amount of dye adsorbed per gram of adsorbent (qe) was calculated as follows:

$$q_{e} = \frac{v}{m} \left( C_{e} - C_{o} \right) \tag{2}$$

Where  $C_o$  and  $C_e$  are initial and equilibrium dye concentrations, respectively (mg L<sup>-1</sup>), V is dye solution volume (L); m is the mass of adsorbent (g).

The effect of adsorption time on the dye removal at various predetermined intervals from (10-120 min) using spectrophotometer UV-VIS double beam PC scanning for measurement of concentration at  $\lambda_{max}$ = 664 nm for methylene blue and  $\lambda_{max}$ = 425 nm for tartrazine was monitored by shaking the reaction mixture and analyzed for the dye content at the end of each contact time. The removal efficiency was determined to study the effects of contact time, initial concentration of dye solution and pH, adsorbent dosage, temperature and agitation rate.

# 3. Result and discussion 3.1. Effect of contact time

The influence of the contact time on the adsorption capacity of two dyes by activated carbon was conducted through batch experiments to achieve the equilibrium as shown in Fig. 1. The mechanism of colour removal can be described by the migration of the dye molecule from the solution to the adsorbents particle and diffusion through the surface. The results showed that equilibrium was reached within 90 min of operation for activated carbon. The adsorption capacity was constant thereafter in the case of both adsorbate observed.



Figure 1. Effect of contact time on the percent removal of MB (20 ppm) and TZ(100 ppm) with activated carbon,.

## 3.2. Effect of initial concentration

The influence of the initial concentration of Methylene blue (5-20 mg  $l^{-1}$ ) and Tartrazine (25-100 mg  $l^{-1}$ ) on the removal efficiency using activated carbon are shown in Figures 2 and 3. The experiments were carried out at fixed adsorbent dose (0.5 g), at room temperature (25 ±1 °C), neutral pH (7.0) and 100 rpm agitation speed.

The effect of the initial dye concentration depends on the immediate relation between the dye concentration and the available binding sites on an adsorbent surface [13]. Fig.2 shows the effect of initial dye concentration. Generally the percentage of dye removal decreases with an increase in initial dye concentration, which may be due to the saturation of adsorption sites on the adsorbent surface[10]. At low concentration, there will be unoccupied active sites on the adsorbent surface, and when the initial dye concentration increases, the active sites required for adsorption of the dye molecules will disappear[14]. However, the increase in the initial dye concentration will cause an increase in the loading capacity of the adsorbent and this may be due to the high driving force for mass at a high initial dye concentration [15]. In other words, the residual concentration of dye molecules will be higher for higher initial dye concentrations. In the case of lower concentrations, the ratio of initial number of dye molecules to the available adsorption sites is low and subsequently the fractional adsorption becomes independent of initial concentration



Figure 2. The influence of initial MB concentration on the dye removal efficiency.

### 3.3. Effect of adsorbent mass

The effect of Activated Carbon (AC) amount on the uptake of the dye was measured for dye concentration 10 mg l<sup>-1</sup> of MB and 100 mg l<sup>-1</sup> of TZ, different shaking time (10-120 min) and different quantity of AC (0.2,0.5, 1 and 1.5 g) at pH = 7 and room temperature ( $25 \pm 1$  °C). The results are shown in Figure 4 and 5. In general, it was found that by increasing the amount of the adsorbent the adsorption rate increased. This increase was most significant when the amount of adsorbent increased from (0.5-1.5 g). Maximum dye removal was achieved within 60-90 minutes after which a decrease in dye concentration was negligible. Increase in dye removal percentage with adsorbent dose can be attributed to increased adsorbent surface area and availability of more adsorption sites. At the beginning of the process the rate of dye removal by the AC was fast during the first 30 min and then decreased gradually.



Figure 3. The influence of initial TZ concentration on the removal efficiency with



Figure 4. The influence of amount of AC on the removal efficiency of MB



Figure 5. The influence of amount of AC on the removal efficiency of TZ

## 3.4. Effect of pH

The pH value of the solution is an important parameter for the adsorption processes, and the initial pH value of the solution has significant influence compared to than the final pH (Hem et al., 2007). To study the effect of pH on MB and TZ adsorption, the experiments were carried out at 10 mg l<sup>-1</sup>initial dye concentration of MB and 100 mg l<sup>-1</sup> with 0.5 g adsorbent dosage at ( $26 \pm 1$  °C). In general, initial pH value may enhance or depress the uptake. This is attributed to the charge of the adsorbent surface with the change in pH value. Figure 5 and 6 show the relationship between the pH value and the removal of MB and TZ.

It can be seen from the Figure 6 of the MB removal that as the solution pH increases, the removal increases. Increasing solution pH increases the number of hydroxyl groups thus, increases the number of negatively charge sites and enlarges the attraction between MB dye and adsorbent surface[13, 16].



Figure 6. The influence of pH on the removal efficiency of MB

Khan (2009), used fly ash as low-cost adsorbent for the removal of Methylene Blue, Malachite Green and Rhodamine B dyes from textile wastewater. In their work they found that, the adsorption of dyes increases from 0.426 to 0.467, 0.232 to 0.394 and 0.286 to 0.367 mg g<sup>-1</sup> for methylene blue, malachite green and Rhodamine B, respectively as the pH is increased from 3 to 9 [17]. Figure 7 shows that as the pH increased the removal efficiency of the TZ removal decreased. This may be attributed to the fact that the adsorption of TZ dye which contain OH functional group is favored by the decrease in pH. Therefore, adsorption may be due to hydrogen bonding, van der Waal forces, and others.

The variation of removal efficiency with pH can be explained by considering the difference in the structure of the two dyes.



Figure 7. The influence of pH on the removal efficiency of TZ with

Proceeding of the 7 <sup>th</sup> ICEE Conference 27-29 May 2014	CEED-2	8/13
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### **3.5. Effect of Agitation Speed**

In a liquid adsorption system, the mass transfer rate of a solute to a particle is affected by liquid film thickness surrounding the particle and the film thickness depends on agitation speed. A series of experiments at different degrees of agitation (from 25 to 125 rpm) were undertaken for the adsorption of MB and TZ on activated carbon as shown in Figures 8 and 9. The results indicate that the degree of agitation influences the removal efficiency as the agitation rate increases from 25 to 125 rpm. At agitation rates higher than 100 rpm the removal efficiency differs only to a quite small extent for MB adsorption, indicating that the film thickness has insignificant effect when the agitation rate is higher than 100 rpm. Hence, an agitation rate of 100 rpm was selected for all the experiments.



Figure 8. The influence of agitation speed on the removal efficiency of MB.



Figure 9. The influence of agitation speed on the removal efficiency of TZ

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### 3.6. Adsorption isotherm

For solid–liquid adsorption systems, adsorption isotherms are important models for the description of adsorption behavior. When the adsorption reaction reaches equilibrium state, the adsorption isotherm can indicate the distribution of dye molecules between the solid phase and the liquid phase[18]. It is significant for understanding the adsorption behavior to identify the most appropriate adsorption isotherm model. In this paper, Langmuir and Freundlich isotherms were employed to investigate the adsorption behavior. Adsorption isotherms were studied at 298 K.

### **3.6.1.** Langmuir isotherm

Langmuir isotherm is tested on the assumption that adsorption occurs at specific homogenous sites within the adsorbent. Once an adsorbate molecule occupies a site, no further adsorption can take place. Thus, an equilibrium value can be reached and the saturated monolayer curve can be express in the equation below which has been successful for the explanation of monolayer adsorption. The linear form of Langmuir equation is given as:

as:  

$$\frac{c_{\varepsilon}}{q_{\varepsilon}} = \frac{1}{q_{\max K_L}} + \frac{c_{\varepsilon}}{q_{\max}}$$
(3)

where  $q_{max}$  is the maximum or monolayer adsorption capacity of the adsorbent (mgg<sup>-1</sup>) and  $K_L$  is the Langmuir adsorption constant (Lmg<sup>-1</sup>), which is related to the free energy of adsorption. Plots of  $C_{e'}/q_{e}$  against  $C_{e}$  at different temperatures are shown in Figures 10 and 11. The maximum adsorption capacity,  $q_{max}$ , and Langmuir constant,  $K_L$  were calculated from the slopes and intercepts of the plots respectively. Values obtained for the adsorption of MB and TZ onto the adsorbent are presented in Table 1.



Figure 10. Langmuir adsorption isotherm of MB-activated carbon adsorption at 25 <sup>o</sup>C. Agreement of the experimental data with Langmuir isotherm model indicates the homogeneous nature of activated carbon surface, i.e. each dye molecule/activated carbon adsorption has equal adsorption activation energy. The results also demonstrate the formation of monolayer coverage of dye molecule at the outer surface of activated carbon. Similar observation was reported by the adsorption of acid orange 10 dye onto activated carbons prepared from agricultural waste bagasse[19] and by the adsorption of direct dyes on activated carbon from coir pith[21].



Figure 11. Langmuir adsorption isotherm of TZ-activated carbon adsorption at 25 <sup>o</sup>C.

It can be predicted whether an adsorption system is favourable or unfavourable using the essential characteristic of the Langmuir isotherm expressed by means of  $R_L$ , a dimensionless constant referred to as separation factor or equilibrium parameter defined by[4]:

$$R_L = \frac{1}{1 + \kappa_L c_0} \tag{4}$$

, where Co is the highest initial concentration. This parameter suggests the type of isotherm to be irreversible ( $R_L = 0$ ), favourable ( $0 < R_L < 1$ ) or unfavourable ( $R_L > 1$ ). It can be seen from Table 1, the value of  $R_L$  is less 1 which suggests that adsorption is favourable.

#### **3.6.2.** Freundlich isotherm

The Freundlich isotherm model is used to describe heterogeneous adsorption process i.e adsorption which takes place on a heterogeneous surface through a multilayer adsorption mechanism. Freundlich isotherm is expressed by the equation

$$q_{e} = K_{f} C_{e}^{1/n}$$
(5)  
This can be linearized to  

$$\log q_{e} = \log K_{f} + \left(\frac{1}{n}\right) \log C_{e}$$
(6)

where  $q_e$  is the amount adsorbed at equilibrium (mg g<sup>-1</sup>),  $C_e$  the equilibrium concentration of the adsorbate (MB and TZ) and  $K_F$  and *n* are Freundlich constants, *n* giving an indication of how favorable the adsorption process and  $K_F$  (mg g<sup>-1</sup>(1 mg<sup>-1</sup>)<sup>n</sup>) is the adsorption capacity of the adsorbent.  $K_F$  can be defined as the adsorption or distribution coefficient and represents the quantity of dye adsorbed onto activated carbon adsorbent for a unit equilibrium concentration. The slope 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[6, 17]. A value for 1/n below one indicates a normal Langmuir isotherm while 1/n above one is indicative of cooperative adsorption [23]. The plot of log  $q_e$  versus log  $C_e$  gives straight lines with slope '1/n' (Fig. 12 and 13), which shows that the adsorption. The Freundlich parameters

Proceeding of the 7 <sup>th</sup> ICEE Conference 27-29 May 2014 CEED	-2 1	1 / 13
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and correlation coefficients ( $\mathbb{R}^2$ ) evaluated from the plots are listed in Table 1. Accordingly, the Freundlich constants ( $K_F$  and n) and  $\mathbb{R}^2$  presented in Table 1, show that Freundlich model could also be used to explain adsorption process



Figure 12. Freundlich adsorption isotherm of MB-activated carbon adsorption at 25 °C.



Figure 13. Freundlich adsorption isotherm of TZ-activated carbon adsorption at 25 °C.

Proceeding of the 7 <sup>th</sup> ICEE Conference 27-29 May 2014 CH	ED-2	12 / 13
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Madala	Parameters	Dye		
Iviodeis		Methelene Blue (MB)	Tartrazine (TZ)	
	$q_{max}(mgg^{-1})$	3.78	21.8	
Langmuir	$K_L$ (Lmg <sup>-1</sup> )	3.3	0.08	
isotherm	$R_L$	0.015	0.11	
	$R^2$	0.99	0.99	
Freundlich isotherm	$K_{\rm F}({\rm mg~g}^{-1}({\rm l~mg}^{-1})^{\rm n})$	0.39	0.38	
	1/n	0.31	0.57	
	$R^2$	0.99	0.97	

Table 1. Isotherm parameters for the adsorption of Methylene blue (MB) and Tartrazine (TZ) on activated carbon.

The correlation coefficient values ( $R^2 > 0.99$ ) (Table 1) show strong positive correlation indicating that adsorption follows Langmuir isotherm. The maximum adsorption capacity,  $q_{max}$ , obtained from the Langmuir plot is 3.78 and 21.8 mgg<sup>-1</sup> for MB and TZ, respectively. This value is higher than those reported by other researchers on to polymeric materials. For instance Malana et al. [22], reported adsorption capacity of 1.017, 1.875 and 2.610 mgg<sup>-1</sup> for methylene blue onto three novel polymeric gels.

# 4. Conclusion

Activated carbon is a promising adsorbent for removal of the basic and azo- dyes. The removal of methylene blue and tatrazine from wastewater using activated carbon has been investigated under different experimental conditions in batch mode. The adsorption of dye was dependent on adsorbent dose, agitation speed and dyes concentration in wastewater. Initial pH of solution affected the adsorption of this dye. The optimum pH for the removal of methylene blue from aqueous solution under the experimental conditions used in this work was 10 while for Tartrazine was 3. Maximum dye removal was observed within 90 min from the beginning of each experiment. Adsorption data was observed to follow Langmuir and Freundlich isotherm models at the studied temperature.

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