



## Preparation of activated carbon from date palm trunks for removal of eosin dye

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### ABSTRACT

Date palm trees are abundant and cheap natural resources in Saudi Arabia. In this study, an activated carbon was prepared from palm trunks by chemical processes. The chemical activation was performed by impregnation of the raw materials after grinding with  $H_3PO_4$  solution (63%), followed by placing of the sample solution on a muffle furnace at 400 °C for 30 min, and then at 800 °C for 10 min. The morphology of the fabricated material was checked using scanning electron microscopy that showed the rough surfaces on the carbon samples. The use of fabricated activated carbon for removal of eosin dye from aqueous solutions at different contact time, initial dye concentration, pH and adsorbent doses was investigated. The experimental results show that the adsorption process attains equilibrium within 20 min. The adsorption isotherm equilibrium was studied by means of the Langmuir and Freundlich isotherms, and it was found that the data fit the Langmuir isotherm equation with maximum monolayer adsorption capacity of 126.58 mg g<sup>-1</sup>. The results indicated that the home made activated carbon prepared from palm trunks has the ability to remove eosin dye from aqueous solution and it will be a promising adsorbent for the removal of harmful dyes from waste water.

### Keywords

Activated carbon; Date palm trunks;  $H_3PO_4$  activation; Adsorption; Dye removal; Eosin dye; Isotherm.

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## 1. INTRODUCTION

Dyes are commonly utilized for coloring textiles, wool, and other materials. These dyes are from coal-tar based hydrocarbons such as benzene, naphthalene, anthracene, toluene, and xylene [1]. These pollutants are difficult to decolorize because of their complex aromatic structure and synthetic origin [2]. Although the exact number of the produced dyes in the world is not known, there are estimated to be more than 100,000 different available dyes. Many of them are known to be toxic or carcinogenic [2-5]. Commonly, these dyes are discharged in the environment in form of colored waste water by many industries without any prior treatment [2].

Purification of waste water from dyes is becoming increasingly important. There are several methods that have been utilized for the removal of dyes from waste water. For example, adsorption [6-8], nanofiltration [9], ozonation [10], and electroflotation [11]. All of these processes have different capabilities for removing dyes. Among these methods, adsorption has been found to be the best technique for waste water purification. The reasons for these are the simplicity of the procedure, ease of operation, and insensitivity of toxic substances [11].

Activated carbon (AC) is commonly the most popular adsorbent. It has the ability to adsorb many dyes with high capacity because of its high surface area [12-15]. However, its application is restricted because it is very expensive and the price of regeneration of the activated carbon is expensive due to the difficulty of desorption of the dye molecules from the activated carbon [16]. Therefore, many studies have focused on preparation of activated carbon from cheaper and renewable raw materials, which are commonly an agricultural byproduct [17]. The raw materials that have been used for fabrication of activated carbon are rice hull [18], olive cake [19], corncob [20], coconut husk [21], rattan sawdust [22], and bagasse bottom ash [23].

Generally, fabrication of activated carbons involves two steps: pyrolysis, and physical and/or chemical activation [24]. In terms of the chemical activation, it involves using dehydrating chemical agents such as  $ZnCl_2$ ,  $H_3PO_4$ , and  $H_2SO_4$ . The most common preferred chemical agent is  $H_3PO_4$  because it is recoverable and the temperature of the activation is relatively low (around 400-500 °C) [17, 25].

The main objective of this study is to investigate the preparation of activated carbon from the trunks of date palms by  $H_3PO_4$  activation. This plant is inexpensive and widespread throughout Saudi Arabia and other parts of the world, such as United States of America, the Canary Islands, northern Africa, the Middle East, Pakistan, and India. In addition, this contribution is investigating removing of eosin dye from aqueous solution in order to evaluate the capacity of the prepared activated carbon. The equilibrium data of adsorption studies were processed to understand the adsorption mechanism of the dye molecules onto the activated carbon.

## 2. Experimental

### 2.1. Chemicals and Materials

Date palm trunks were collected from a local garden near Taif, KSA. Whatman filter paper (diam. 15 mm) was purchased from Sigma-Aldrich (Nottingham, UK). Eosin dye (99%) was purchased from Sigma-Aldrich (Nottingham, UK) and used without further purification. Distilled water was employed for preparing all the solutions and reagents.

### 2.2. Instrumentation

A pH meter (Fisherman Hydrus 300, Thermo Orion, Beverly, MA, USA), a hot plate-stirrer from VWR International LLC (West Chester, PA, USA), a scanning electron microscope (SEM) Cambridge S360 from Cambridge Instruments (Cambridge, UK), UV-Vis spectrophotometer from Thermo Scientific™ GENESYS 10S (Toronto, Canada), a furnace (WiseTherm high temperature muffle furnaces, Wisd Laboratory Instrument, Wertheim, Germany).

### 2.3. Preparation and Characterization of Activated Carbon

Figure 1 illustrates the steps that were used for fabrication of the activated carbon from date palm trunks. The collected palm trunks were washed with distilled water, dried in sunlight for 24 h, and ground. The dried palm trunks powder was sieved to 250-500  $\mu m$  in size. Then, it was soaked with a boiling solution of 63%  $H_3PO_4$ . After 24 h, the excess amount of solution decanted off and air dried. The material was carbonized at 400 °C for 30 min using a muffle furnace. The dried material was activated in a muffle furnace at 800 °C for 10 min. Subsequently, the carbonized material produced was taken out and washed with plenty of distilled water to remove the residual acid. It was dried at 60 °C for 24 h and stored. The surface morphology of prepared material was observed by scanning electron microscopy (SEM), and the sample was gold coated prior to SEM observation.



Fig 1: The process carried out on fabrication of activated carbon from date palm trunks.

## 2.4. Removing of Eosin Dye From Aqueous Solution

### 2.4.1. Effect of Contact Time and Dye Concentration

A stock solution of eosin dye of  $20 \text{ mg L}^{-1}$  was prepared, which was diluted to the required initial concentrations. Adsorption isotherms were performed in a set of 43 Erlenmeyer flask (250 mL). The effect of contact time on removal of eosin dye was studied for a period of 50 min. The adsorption experiments were carried out by agitating 0.2 g of adsorbent (activated carbon fabricated from palm trunk) with different concentrations of eosin dye ( $2, 5, 10, \text{ and } 20 \text{ mg L}^{-1}$ ) at different conical flask and kept in an isothermal shaker at 200 rotation per minute (200 rpm), for each of the different contact times (5, 10, 15, 20, 30, 40, and 50 min). The flasks were then removed from the shaker and the sample solutions were filtered using filter paper before analysis to minimize the interference of the carbon fines with the analysis. The



performance of the fabricated activated carbon in terms of removing of dye from aqueous solution was checked by monitoring the absorbance changes at a wavelength of maximum absorbance (517 nm) using a UV-Vis spectrophotometer [16]. The amount of adsorbed eosin dye was calculated using the following mass balance equation:

$$q_{eq} = \frac{(C_i - C_{eq})V}{1000 m} \quad [26]$$

Where  $q_{eq}$  is the amount of dye adsorbed ( $\text{mg g}^{-1}$ ),  $C_i$  is the initial dye concentration ( $\text{mg L}^{-1}$ ),  $C_{eq}$  is the equilibrium dye concentration ( $\text{mg L}^{-1}$ ),  $V$  is the volume of solution (mL), and  $m$  is the mass of activated carbon sample (mg).

#### 2.4.2. Effect of Adsorbent Dosage

Different dosages of the adsorbents (0.2, 0.4, 0.6, 0.8, and 1g) were added in different conical flasks containing 50 mL of the dye ( $10 \text{ mg L}^{-1}$ ), agitated in a shaker for 50 min at a speed of 200 rpm at a room temperature of  $25 \text{ }^\circ\text{C}$ . The content of each flask was then filtered and analyzed after the agitation time.

#### 2.4.3. Effect of pH

The effect of the solution pH on eosin dye removal was investigated similarly as described above by changing the initial pH (1-11), and the pH was adjusting by 0.1 M HCl solution or 0.1 M NaOH solution. For this particular study, 50 mL of eosin dye ( $10 \text{ mg L}^{-1}$ ) was added to 0.6 g of the activated carbon into different 250 mL conical flask and agitated at 200 rpm for 50 min. Each experiment was duplicated under identical conditions. Langmuir and Freundlich isotherms were employed to study the adsorption capacity of the adsorbent.

### 3. RESULTS AND DISCUSSION

#### 3.1. Fabrication of The Activated Carbon

The purpose of this study was to investigate fabrication of activated carbon from available raw materials. In this study, the activated carbon was fabricated from palm trunks since the date palms are abundant and inexpensive natural resource in Saudi Arabia. The palm trunks-based activated carbon was fabricated using activating agents that was phosphoric acid solution. The reason for choosing this activated agent is that phosphoric acid solution can produce activated carbon with high porosity [27, 28]. The morphology of the fabricated activated carbon was studied using SEM instrument. Figure 2 shows the appearance of the fabricated activated carbon using different magnifications. The SEM micrographs indicated that the fabricated material had cavities, and rough surfaces on the carbon samples. These micrographs confirmed that the fabricated material had high porosity, which can hold more solute from the solution during adsorption [28].

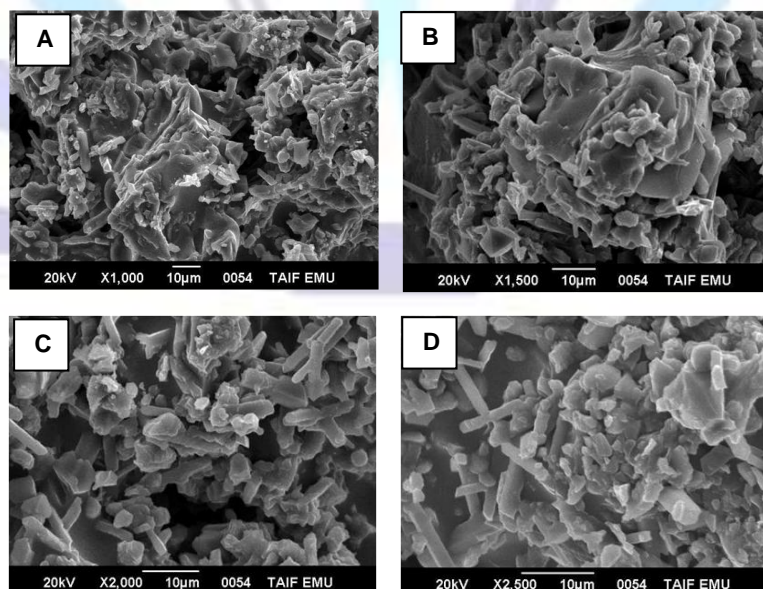
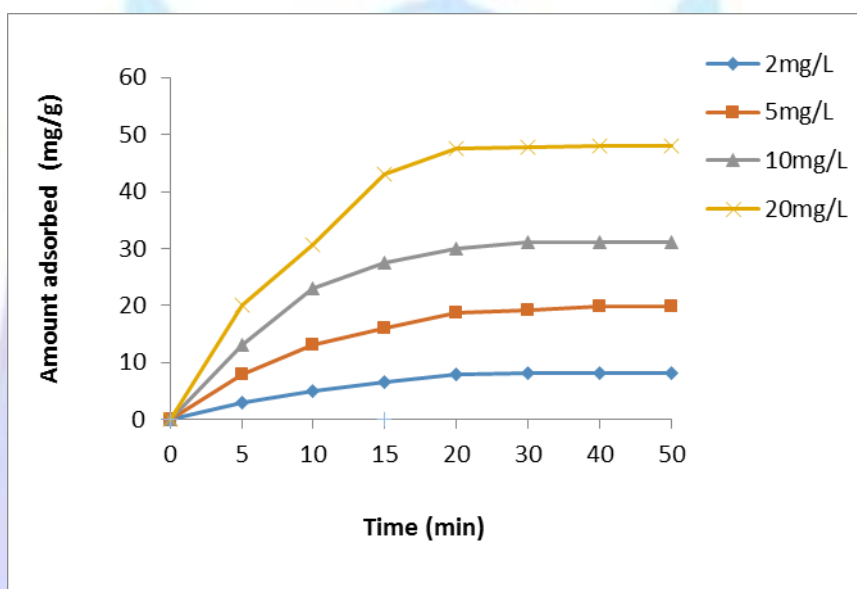


Fig 2: SEM micrographs of the fabricated activated carbon using different magnifications: (A) 1000 ×, (B) 1500 ×, (C) 2000 ×, and (D) 2500 ×.

### 3.2. Effect of Contact Time and Dye Concentration on Adsorption of Eosin Dye

In this study, eosin dye was used in order to check the ability of the prepared activated carbon to remove dyes from aqueous solution. Eosin dye is the most commonly used material for dyeing cotton, wood, and silk due to its vivid colour [29]. The most important parameter such as contact time, initial concentration, sorbent dosage, and pH on adsorption of eosin dye were studied.

The effect of agitation time and concentration of dye on adsorption was investigated. A series of contact time experiments for eosin dye have been carried out at different concentrations ( $2\text{--}20\text{ mg L}^{-1}$ ) and at room temperature of  $25\text{ }^{\circ}\text{C}$ , at intervals of 50 min. 50 mL of dye was mixed with 0.2 g of the activated carbon, followed by filtration of the solution using the filter paper. The performance of the activated carbon was examined using UV-Vis spectrophotometer at 517 nm. The relationship between contact time and the percentage removal of eosin dye from aqueous solution with activated carbon produced from palm trunks is shown in figure 3. From the obtained results, it can be seen that the removal of dye increased as contact time increase. With increase in contact time, the external mass transfer coefficient increase, resulting in quicker adsorption of the dye molecules by the activated carbon. However, the adsorption capacity gradually increases until equilibrium is reached at 20 min. At this point, the amount of eosin dye adsorbed onto the adsorbent (activated carbon) is in state of dynamic equilibrium with the amount of the dye desorbing from the adsorbent. The time required to attain this state of equilibrium is called the equilibrium time. The amount of eosin dye adsorbed at the equilibrium time reflects the maximum adsorption capacity of the adsorbent under those operating conditions. A similar trend has been reported in literature [16, 30]. The adsorption density at equilibrium increased with increase in the initial dye concentration from 2 to  $20\text{ mg L}^{-1}$  from 8 to  $47\text{ mg g}^{-1}$ .



**Fig 3: The variation of adsorption capacity with contact time at various initial dye concentration at  $25\text{ }^{\circ}\text{C}$  (adsorbent= 0.2 g, V= 50 mL).**

The SEM images of the activated carbon after adsorption of eosin dye molecules were studied. Figure 4 shows the SEM micrographs of the activated carbon after adsorption of eosin dye ( $10\text{ mg L}^{-1}$ ) and the agitation time was 50 min. From the images, it can be seen that the microstructure of the prepared activated carbon was changed and the cavities and roughness surface of the activated carbon were decreased due to the adsorption of the dye on the surface of the activated carbon. This investigation showed that palm trunks can be an effective material for preparation of activated carbon in order to use it as an adsorbent for removing of eosin dye from aqueous solution.

### 3.3. Effect of Adsorbent Dosage

The effect of the activated carbon dosage on removal of dye was investigated. In this study, adsorbent dosage was varied from 0.2 to 1 g, under the specific conditions (50 mL of eosin dye ( $10\text{ mg L}^{-1}$ ), contact time of 50 min, 200 rpm shaking speed, and at room temperature of  $25\text{ }^{\circ}\text{C}$ ). Figure 5 shows the effect of adsorbent dosage on removal of eosin dye by the fabricated activated carbon. From the figure, it can be seen that the adsorbance of the eosin dye increased as the adsorbent dosage increased, which is due to the increase in adsorbent surface area of the adsorbent [1].

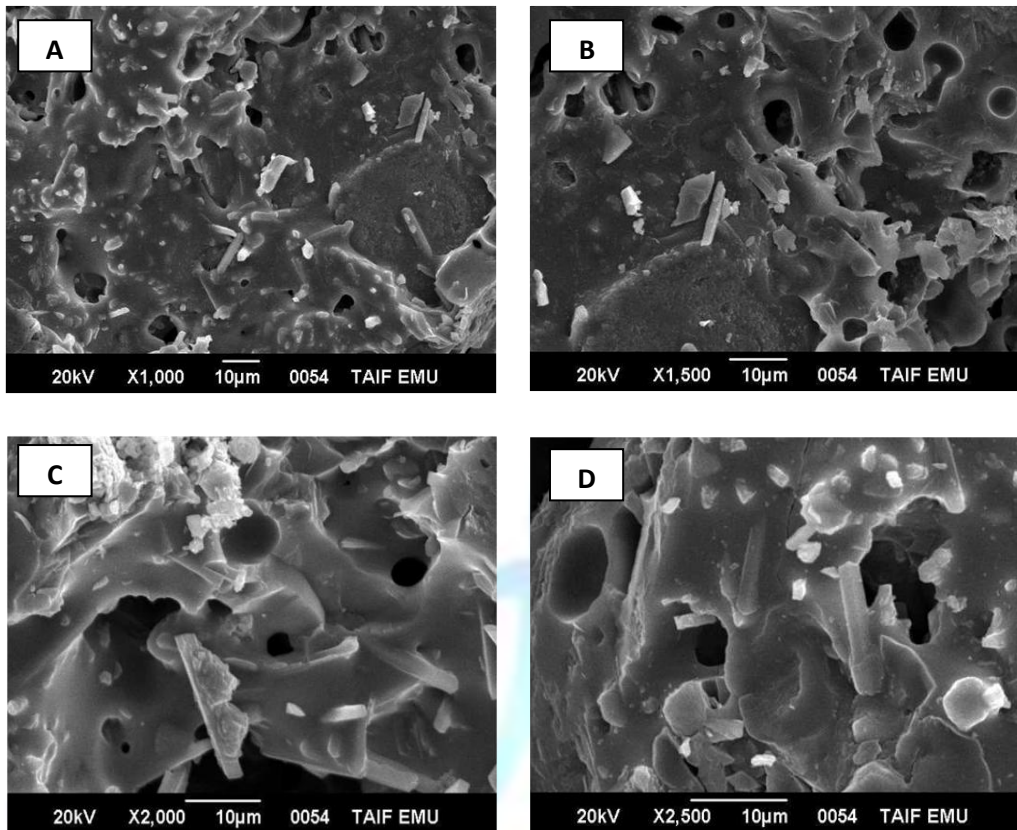


Fig 4: SEM micrographs of the fabricated activated carbon after adsorption of eosin dye molecules using different magnifications: (A) 1000 ×, (B) 1500 ×, (C) 2000 ×, and (D) 2500 ×.

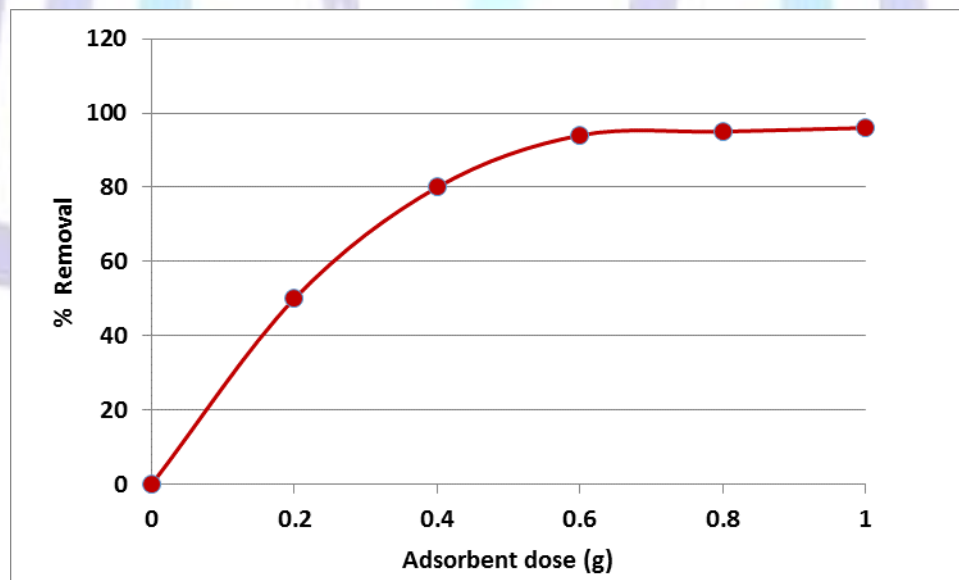


Fig 5: Effect of adsorbent dosage on removal of eosin dye; agitation time (50 min), 50 mL of eosin dye ( $10 \text{ mg L}^{-1}$ ).

### 3.4. Effect of pH

The pH of the dye solution plays an important role in the adsorption process especially on adsorption capacity [6]. In this study, the adsorption of eosin dye was tested in different pH values 1-11 using dilute HCl or NaOH solutions with keeping other parameters constant. The effect of pH on the adsorption of eosin dye by the fabricated activated carbon is presented in figure 6. From the figure, it can be seen that the pH of the solution affects the amount of dye adsorbed. At lower pH, the surface of the adsorbent becomes positively charged due to the high concentration of  $H^+$  ions, which enhances the adsorption of the negatively charged eosin dye (anionic dye) through electrostatic forces attraction. In contrast, in alkaline conditions the surface of the adsorbent is negatively charged due to the high concentration of  $OH^-$  ions on the surface of the adsorbent competing with the eosin dye for adsorption sites. A similar trend was observed for adsorption of eosin dye onto fly ash [16].

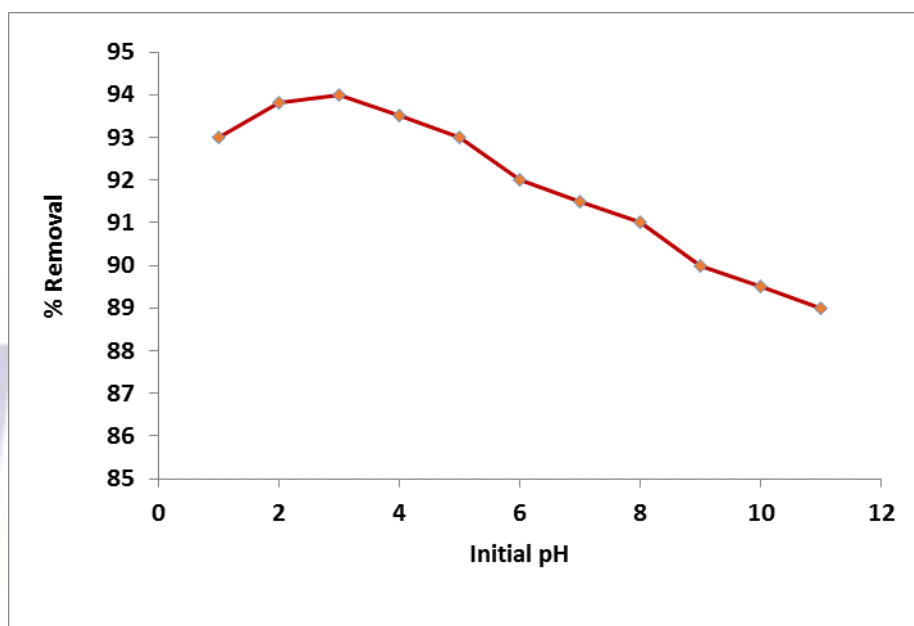


Fig 6: Effect of pH on removal of eosin dye by the prepared activated carbon from palm trunks, adsorbent dose (0.6 g) agitation time 50 min; 50 mL of eosin dye ( $10 \text{ mgL}^{-1}$ ).

### 3.5. Adsorption Isotherms Studies

The adsorption isotherm describes how adsorbates interact with adsorbents. The equilibrium adsorption isotherm can be graphically depicted by plotting a solid phase against a liquid phase concentrations. Several isotherm equations are available. In this study, the adsorption equilibrium data of eosin dye onto the palm trunk-based activated carbon were analyzed in terms of Langmuir and Freundlich isotherm models. The applicability of the isotherm equation is compared by judging the correlation coefficients ( $R^2$ ) [30].

#### 3.5.1. Langmuir Adsorption Isotherm

Langmuir adsorption isotherm assumes that the adsorption of adsorbates occur on specific homogeneous surface by monolayer adsorption without any interaction between adsorbed species. The Langmuir adsorption isotherm equation as the following equation:

$$\frac{C_{eq}}{q_e} = \frac{1}{Kb} + \frac{C_{eq}}{b} \quad [26]$$

Where  $C_{eq}$  is the dye concentration remaining in the solution at equilibrium ( $\text{mg L}^{-1}$ ),  $q_e$  is the amount of solute adsorbed per unit weight of adsorbent ( $\text{mg g}^{-1}$ ),  $K$  is the maximum adsorption capacity corresponding to complete monolayer coverage ( $\text{mg g}^{-1}$ ), and  $b$  is the Langmuir isotherm constant ( $\text{L mg}^{-1}$ ). A plot of  $\frac{C_{eq}}{q_e}$  versus  $C_{eq}$  gave a straight line with a slope  $\frac{1}{Kb}$  and an intercept of  $\frac{1}{b}$  (figure 7). From the figure, it can be seen that the adsorption of the eosin dye on the activated carbon follows the Langmuir isotherm. Values of  $K$  and  $b$  were calculated from the slope and intercept of the linear plots and presented in Table 1. The applicability of the Langmuir isotherm suggests the monolayer coverage of the dye on the surface of the palm trunk-based activated carbon.



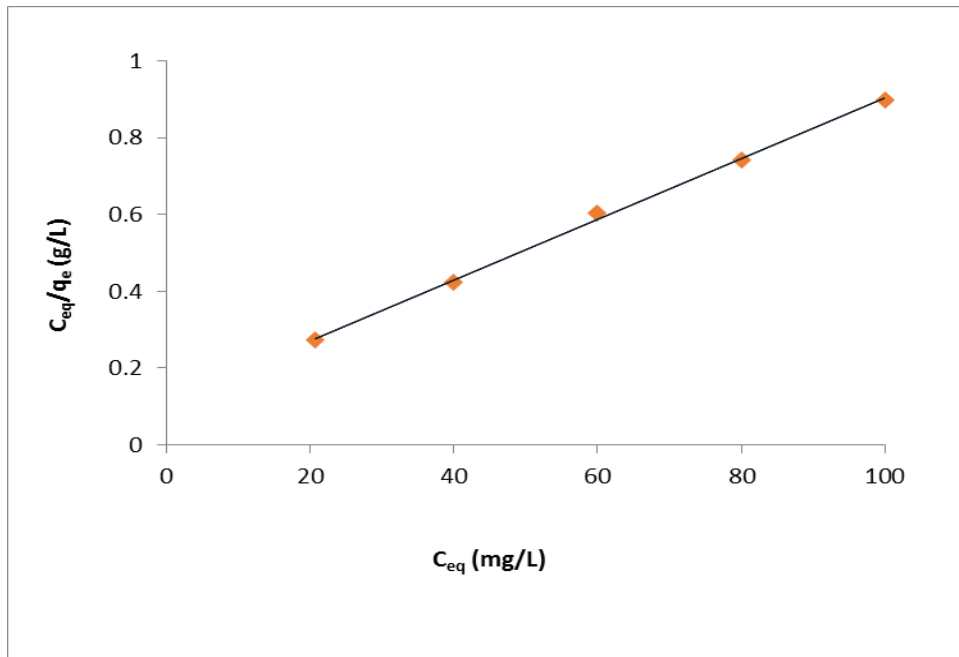


Fig 7: Plot of Langmuir adsorption isotherm of eosin dye onto powdered activated carbon.

The essential characteristic of the Langmuir isotherm can be expressed by a dimensionless constant called equilibrium parameter ( $R_L$ ), which can be defined by:

$$R_L = \frac{1}{(1+bC_e)} \quad [7]$$

Where  $b$  is the Langmuir isotherm constant ( $L \text{ mg}^{-1}$ ),  $C_e$  is the initial dye concentration ( $\text{mg L}^{-1}$ ), and  $R_L$  value indicates the type of isotherm.  $R_L$  can be either unfavourable if  $R_L > 1$ , linear if  $R_L = 1$ , favourable if  $0 < R_L < 1$ , and irreversible if  $R_L = 0$  [30, 31]. As seen from Table 1, the  $R_L$  values were found to be between 0 and 1 for dye concentrations of 2, 5, 10, and 20  $\text{mg L}^{-1}$ . In this work, the maximum monolayer coverage capacity ( $K$ ) from Langmuir isotherm was calculated to be  $126.58 \text{ mg g}^{-1}$ , ( $b$ ) was  $0.069 \text{ L mg}^{-1}$ , the  $R^2$  value is 0.998 indicating a very good mathematical fit and the activated carbon is favorable for adsorption of eosin dye under conditions used in this study.

Table 1: Langmuir isotherm constants for eosin dye.

Dye concentration ( $\text{mg L}^{-1}$ )	$K$ ( $\text{mg g}^{-1}$ )	$b$ ( $\text{L mg}^{-1}$ )	$R^2$	$R_L$
2	126.58	0.069	0.998	0.878
5				0.743
10				0.591
20				0.420

### 3.5.2. Freundlich Adsorption Isotherm

Freundlich adsorption isotherm assumes that the adsorption of adsorbates occurs on heterogenous adsorption surface by multilayer adsorption and that the amount of adsorbate adsorbed increases infinitely with an increase in concentration. The linearized form of Freundlich model is represented by the following equation:

$$\log q_e = \log k_f + \left(\frac{1}{n}\right) \log C_e \quad [26]$$



where  $q_e$  is the amount of eosin dye adsorbed at equilibrium ( $\text{mg g}^{-1}$ ),  $C_e$  is the equilibrium constant of the adsorbate ( $\text{mg L}^{-1}$ ),  $k_f$  and  $\frac{1}{n}$  are Freundlich constants incorporating the factors affecting the sorption capacity and the degree of non-linearity between the dye concentration in the solution and the amount adsorbed at equilibrium, respectively, and they can be obtained from the intercept and slope of  $\log q_e$  versus  $\log C_e$  plot. As can be seen in figure 8, the linear plot of  $\log q_e$  vs  $\log C_e$  indicates that the adsorption of eosin dye also follows the Freundlich adsorption isotherm. The slope ( $\frac{1}{n}$ ) ranging between zero and one is a measure of adsorption intensity or surface heterogeneity and becoming more heterogeneous as its value gets closer to zero. A value for ( $\frac{1}{n}$ ) below 1 indicates a normal Langmuir isotherm while ( $\frac{1}{n}$ ) above 1 is indicative of cooperative adsorption [32]. In this study, the value of  $\frac{1}{n}$  was 0.240 indicating that the adsorption of eosin dye also follows Freundlich isotherm. However,  $R^2$  value of Langmuir isotherm was compared with those of Freundlich isotherm, and it was found that the Langmuir isotherm yields a somewhat better fit ( $R^2 = 0.998$ ) than the Freundlich isotherm ( $R^2 = 0.975$ ).

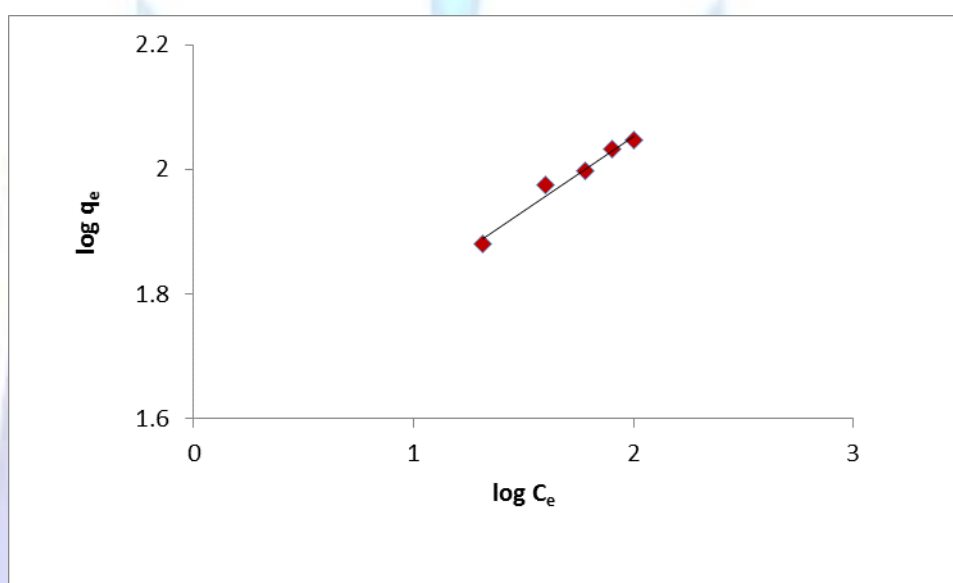


Fig 8: Freundlich isotherm plot of the adsorption of eosin dye onto activated carbon.

#### 4. CONCLUSION

From the results of the present investigation, the home made activated carbon has been fabricated successfully from date palm trunks. Eosin dye had been used to check the performance of the fabricated activated carbon. The results indicated that the fabricated activated carbon from palm trunks has the ability to remove eosin dye from aqueous solution, which was confirmed by UV-Vis spectrophotometer. The equilibrium data followed the Langmuir isotherm and the maximum monolayer adsorption capacities of  $126.58 \text{ mg g}^{-1}$ . In conclusion, palm trunks are a potential and low-cost natural material for the preparation of activated carbon. A future study investigating the removal of dyes from a real sample such as waste water would be interesting.

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