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# Use of agriculture-based waste for basic dye sorption from aqueous solution: Kinetics and isotherm studies

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**Abstract:** Low-cost bare palm branches were prepared as a sorbent for methylene blue dye from aqueous solution. The effect of reaction parameters such as sorbent dose and its particle size, initial dye concentration and the medium temperature were investigated using a batch sorption technique. Additionally, the maximum saturated monolayer sorption capacity of bare palm branches for methylene blue dye was investigated. The isotherm data was well-described by the Freundlich equation. Based on the adsorption capacity, it was shown that the use of bare palm branches was a promising low-cost agriculture waste material for the adsorption of dyes from aqueous solutions. Kinetic parameters of adsorption such as the rate constant and the intra-particle diffusion rate constant were determined. The principal conclusions of the study were that the reaction follows the pseudo-second order reaction kinetics.

**Keywords:** Bare Palm Branches, Sorption, Dye, Isotherm, Reaction Kinetics

## 1. Introduction

In Egypt, industry represents a significant proportion of total wastewater discharges. The volume of water used by industry in Egypt 2000 was estimated to be $6.1\times10^9$ $m^3$, which is expected to be $8.6\times10^9$ $m^3$/year by the year 2025 [1]. Amongst the most highly polluting wastewater sources is the textile and dyeing industry which consumes large volumes of water primarily in the dyeing and finishing operations where dyes are extensively used [2]. The discharge of dye wastewater to receiving waters could potentially degrade water quality and impact on human health due to the toxic and carcinogenic effects of some dyes [3- 5].

Methylene blue dye is a cationic dye discharged to the natural environment as a result of human activities. For instance, it is widely used in colouring paper, temporary hair colouring, dyeing cotton and wools, and coloring of paper stocks. The removal of such dye from any wastewater is of utmost importance due to the serious environmental damage that can occur as a result of contact with it. Even though methylene blue is not considered to be a very toxic dye it can reveal very harmful effects on the living things, particularly in the case of people. After inhale symptoms such as difficulties in breathing, vomiting, diarrhea and nausea can occur in humans [6, 7].

As a result of the total ban on the importation of textile and leather products by the Government of Egypt, there has been increased activity in the local textile, finishing and dyeing industries to meet the demands for textile and coloured products. Large volumes of wastewater are therefore generated during the dyeing and finishing processes which are usually characterized by components high in both colour and organic content [2, 8]. The majority of these dyes are synthetic in nature and are usually composed of aromatic rings in their structure, which makes them carcinogenic and mutagenic [9], inert and non-biodegradable when discharged into waste [10]. Therefore, the treatment of such wastewater containing soluble dyes requires virtually complete removal followed by safe disposal [11].

Sorption of coloured components from aqueous solutions has proven to be an excellent way to treat such effluents and is also a cost effective technique. Several studies have shown that numerous low-cost materials have been successfully applied in the removal of dyes from aqueous solution, some of which are peat for Basic Blue 69 and Acid Blue 25 [10], giant duckweed for Methylene Blue [12], Neem leaf powder for Brilliant Green [13], rice husk for Malachite Green [14], sugar cane dust for basic dyes [15], tree fern for Basic Red 13 [16], and Fuller's earth for Methylene Blue [17]. Mittal, Mittal, and Kurup [18] applied the hen feather for Indigo Carmine removal. In addition, Bulut et al., [19] used bentonite for Congo Red removal. Mittal et al. [20] used activated charcoal for yellow ME 7 GL industrial dye effluent removal. Furthermore, carboxymethyl cellulose, polyvinyl alcohol and chemically treated rice husk biomass was used for the biosorption of Everdirect Orange-3GL and Direct Blue-67 dyes [21]. Additionally, Mittal et al.[22] applied hen feather as a cheap adsorbent material for wastewater effluent contaminated with Congo dye. However, it is noted from the literature there is a lack in the using of the bare palm branches as a cheap sorbent material.

The search for new, readily available and economical sorbents in developing countries, like Egypt, is ongoing to meet the purification needs for the large volume of textile industry effluents produced on a daily basis. One possible suitable sorbent for these wastes is the use of natural fibrous materials and research by Onwuka et al. [23] has shown that there is a considerable volume of household wastes and crop residues that might be suitable. According to their findings, over 66% of the palm kernel and its fibre are burnt, a little above 5% is used as feed for animals and over 29% is left unused in the fields.

This study is aimed at investigating the possibility of using bare palm branches, a local readily available agricultural waste product in Egypt, for the removal of Methylene blue from aqueous solution. This class of dyes is the most problematic, because they tend to pass through conventional treatment systems unaffected. The system variables examined include: sorbent dose, initial concentration of the dye, and temperature. Isotherm studies for the batch experiments described below, and the development of an isotherm model, were undertaken. In addition, the kinetic order was investigated.

# 2. Materials and Methods

### *2.1. Experimental Materials*

A simulated industrial textile wastewater was synthetically prepared using Methylene Blue Dyestuff ($C_{16}H_{18}N_3SCl.3H_2O$; molecular weight: 373.9) supplied by Merck.

Bare palm branches were used as sorbent material to remove the dye. Received bare palm branches from El-Minia city, in the south of Egypt were first washed thoroughly with water to remove all foreign materials, dirt and fibers then cut it to small pieces of about 1 to 2 mm in size which equals 16 to 8 BSS (according to the British standard screens BSS mesh). The cleaned branches are dried at 383 K for 3 hours in a drying oven until it is completely dry by reaching a constant weight. Chemically, the branches after 15.9% moisture content, and it is rich in organic matter (92.99%). Ultimate analysis of branches which is carbon, nitrogen, sulphur, phosphorous and potassium was 53.94%, 0.78%, 0.37%, 0.37% and 0.21%, respectively.

The Brunauer-Emmett-Teller (BET) surface analysis which was carried using an automated adsorption apparatus (Micromeritics Pulse Chemisorbs 2705) shows (9.13 $m^2$/g and 19.6 μm, values) for surface area and pore diameter, respectively.

### *2.2. Experimental Methods*

Palm fibre in contact with 25 ml of dye solutions which were prepared in distilled water at concentrations ranging from 100 to 400 mg/L was used to investigate the equilibrium uptake of dye ions at 303 K with a sorbent mass of 1.0 g (16 BSS, particle size). The solution with the branches was mechanically shaken at 200 rpm and 303 K for 5 hours. Thereafter, the samples were centrifuged at 400 rpm for 15 minutes to separate the sorbent material. Subsequently, the equilibrium concentration of the dye remaining in solution was determined spectrophotometrically after the wavelength of the dye was determined. Moreover, three duplicate of the samples were measured and the average was calculated in each case.

In this study, the adsorption data were analyzed using Langmuir and Freundlich adsorption isotherm models to describe the sorption equilibrium.

The Langmuir isotherm is based on the assumption that maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface. The linear form of the Langmuir isotherm is represented as follows:

$$\frac{C_e}{q_e} = \frac{1}{K_L a_L} + \frac{1}{K_L} C_e \tag{1}$$

where Ce is the equilibrium dye concentration (mg $L^{-1}$), $q_e$ is the mass of dye adsorbed per unit mass of adsorbent (mg $g^{-1}$), $K_L$ and $a_L$ are the Langmuir constants related to the adsorption capacity and rate of adsorption, respectively.

The Freundlich isotherm model is applicable to a highly heterogeneous surface:

$$\ln(q_e) = \ln K_F + \frac{1}{n} \ln C_e \tag{2}$$

where $K_F$ relate to the adsorption capacity of the adsorbent and and n is a measure of the adsorption intensity. The magnitude of the coefficient, 1/n, gives an indication of how favourable the adsorption is; values of $n > 1$ represent favorable adsorption conditions [10].

#### *2.2.1. Kinetic Study*

The experimental set-up is principally consists of air compressor, flow meter and Perspex column (75 mm inside diameter and 600 mm height) and distributor (5 mm thickness and 1.0 mm hole diameter). The column was filled with the dye solution before each run at concentrations ranging from 10 and 40 mg/L and the sorbent masses added were 1, 3, 4 and 5 g (16 BSS, particle size). A gas stirring technique was used to contact the sorbent and the dye solution. The fixed bed column was connected to an air supply system which enabled the air flow rate and temperature to be controlled.

The kinetic studies describe the rate of adsorption and the time to reach equilibrium. Kinetic modelling is useful to optimize wastewater treatment design. Pseudo first order and second order kinetic models were examined, in particular, in the context of methylene blue adsorption by bare palm branches.

### *2.3. Analytical Methods*

The residual dye concentrations of the samples were measured after the reaction time using a spectrophotometer (SHIMADZU-UV 1601, Model TCC-240A).

## 3. Results and Discussions

### *3.1. Preliminary Adsorption Studies*

#### *3.1.1. Effect of Contact time*

The contact time studies were carried out to understand the trend of dye adsorption over a period of time at 40 mg/L dye concentration by taking fixed amount (1.00 g) and particle size (16 BSS) of the bare palm branches. Figure 1 showed the amount of the dye adsorbed per unit mass of adsorbent ($q_t$) increased with increasing time before reaching a plateau. The equilibrium is achieved after a contact time of approximately 4 h. However, to allow some factor of safety at higher dye concentrations, all the experiments were carried out with 5 h of contact time. There was no noticeable improvement in dye removal when the contact time was prolonged. The initial high rate of dye uptake may be attributed to the existence of the bare surface; however, the number of available adsorption sites decreased as the concentration of dye adsorbed increased [24]. This observation of 4 hours contact time was also reported by Mittal et al., (2013) for Eosin dye removal onto De-oiled waste Soya adsorbent [25].

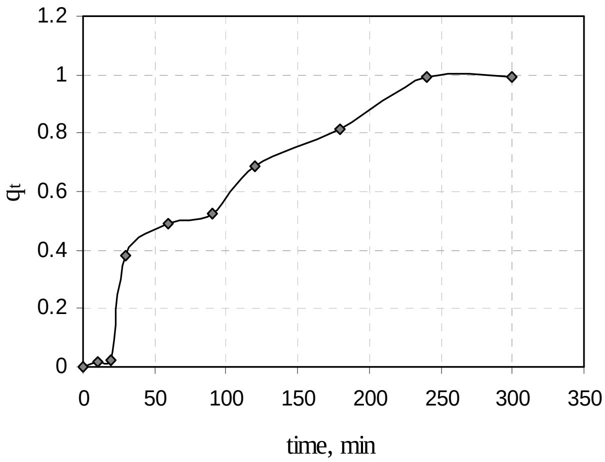


***Figure 1.*** *Effect of contact time on the adsorption of dye by palm branches.*

#### *3.1.2. Effect of Adsorbate Concentration and Temperature*

The adsorption behaviours of methylene blue dye on bare palm branches was examined in concentrations ranging from 100 to 400 mg/L, at a fixed sorbent of 1.00 g of 16 BSS particle size and 303 K. The experimental results reveal that sorption was more favourable for the lower initial dye concentrations than the higher ones. This finding is due to the increase in availability of surface active sites resulting from the increased adsorbent/adsorbate ratio [26]. This result in accordance with Gupta t al., [27] for azo dye removal using $TiO_2$, nevertheless, Mittal and Gupta [28] recorded that the sorption of azo dye Eriochrome Black T onto bottom ash and de-oiled soya is increased with increasing the concentration uptake.

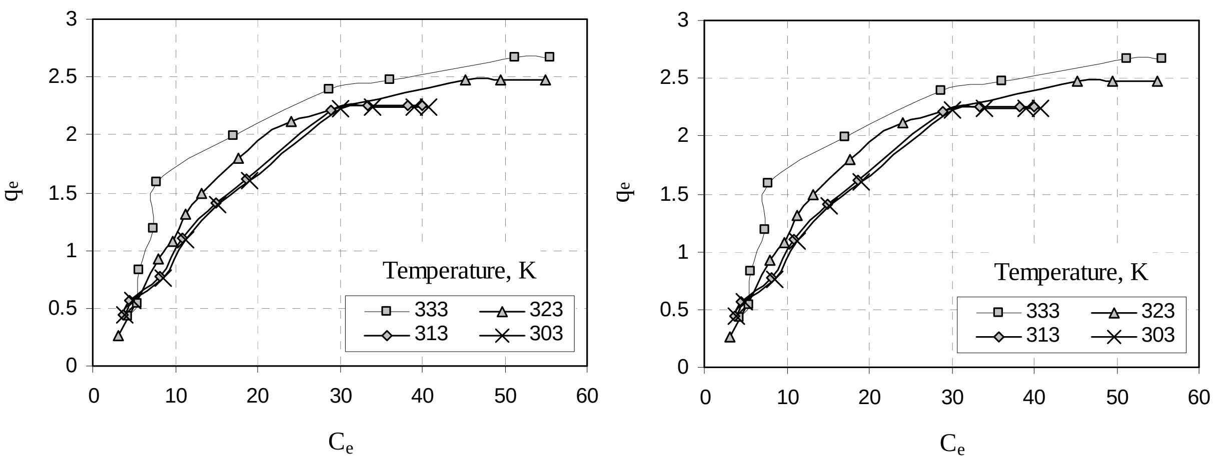


***Figure 2.*** *Effect of temperature variation on the dye adsorption*

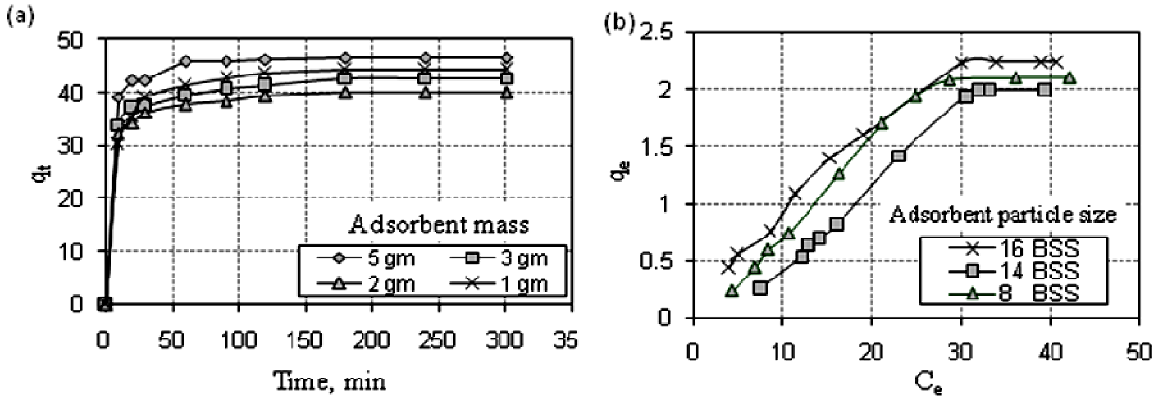


***Figure 3.*** *Effect of sorbent material on sorption process: (a) effect of sorbent mass; (b) effect of sorbent particle size*

The adsorption was studied with initial dye concentration of 40 mg/L, temperatures 303, 343, 353 and 363K, using adsorbent mass of one g (16BSS, particle size) with constant shaking (200 rpm, 5 hr) to illustrate the effect of temperature on the sorption isotherm. Fig. 2 represented that sorption capacity decreased with an increase in temperature. This decrease in sorption capacity with temperature is due to the enhancement of the desorption step in the sorption mechanism indicating that the process is exothermic [29]. It is known that decreasing sorption capacity with increasing temperature is mainly due to the weakening of sorptive forces between the active sites on the palm kernel fibre and anionic dye species, and also between adjacent dye molecules on the sorbed phase. The conventional mechanism of a physisorption system is that an increase in temperature usually increases the rate of approach to equilibrium, but decreases the equilibrium capacity [15]. However, this finding is not in agreement with Mittal et al. [22] who found the reaction is endothermic in the treatment of dyes using de-oiled soya and hen feather, respectively.

#### *3.1.3. Effect of Mass and Particle Size of Sorbent*

The effect of the sorption capacity on the removal of methylene bue dye was examined by varying the sorbent masses of 1.0, 2.0, 3.0 and 5.0 g by fixing other conditions. It is apparent from (Fig. 3a) that by increasing the dose of the bare palm branches, the number of sorption sites available for sorbent interaction is increased, thereby resulting in the increased percentage dye removal from the solution. The decrease in sorbent capacity may be attributed to: (a) the increase in sorbent dose at constant dye concentration and volume leads to unsaturation of sorption sites [30]; (b) particulate interaction such as aggregation resulting from high sorbent dose. Such aggregation would result in a decrease in total surface area of the sorbent and an increase in diffusional path length [30]. Those results of increasing the sorption capacity with increasing the sorbent amount in agreement with the previous findings of Mittal et al. [22] and Mittal, Jhare, and Mittal [25].

Three different particle sizes, 16, 14 and 8 BSS, of bare palm branches (1.00 g of adsorbent, 40 mg/L dye solution concentration) were examined to investigate the effect of particle size on the sorption process (Fig. 3b). The maximum sorption capacity was achieved at 16 BSS particle size. The increase in the particle size may lead to unsaturation of sorption sites and particulate interaction such as aggregation resulting from high sorbent dose [30]. Thus, total surface area of the sorbent is decreased and an increase in the diffusional path length due to the decrease in available surface area. For larger particles, the diffusion resistance to mass transfer is high and most of the internal surface of the particle may not be utilized for adsorption and so the amount of dye adsorbed is relatively small. This phenomenon of increasing the sorption capacity with decreasing the adsorbent size was previously mentioned by Mittal, Jhare, and Mittal [25] for dye sorption with de-oiled waste soya.

### *3.2. Adsorption Isotherms*

The adsorption isotherm is the relationship between the amount of a substance adsorbed and its concentration in the equilibrium solution at a constant temperature. The function isotherm models, for example Langmuir and Freundlich , to the adsorption study was assessed by comparing the correlation coefficients, $R^2$ values.

As shown in Fig. 4 Langmuir isotherm was chosen to show the maximum adsorption capacity. The values of maximum adsorption capacity can be obtained from the slope of the plot of $Ce/q_e$ versus Ce. The correlation coefficient of the Langmuir isotherm, $R^2$ is 0.94. The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless constant as shown in Table 2 for different conditions.

In addition, Freundlich isotherm is applied by plotting of ln qe vers us ln Ce in Fig. 4 shows a straight line with a correlation coefficient of 0.98. The constants $K_F$ and 1/n were determined from the plot and are presented in Table 1.

As seen from Table 1, a high regression correlation coefficient was shown by the Freundlich model, and indicates that the Freundlich model was suitable for describing the sorption equilibrium of dye by bare palm. Similar results were reported for dye adsorption on powder and flakes chitosan [31].

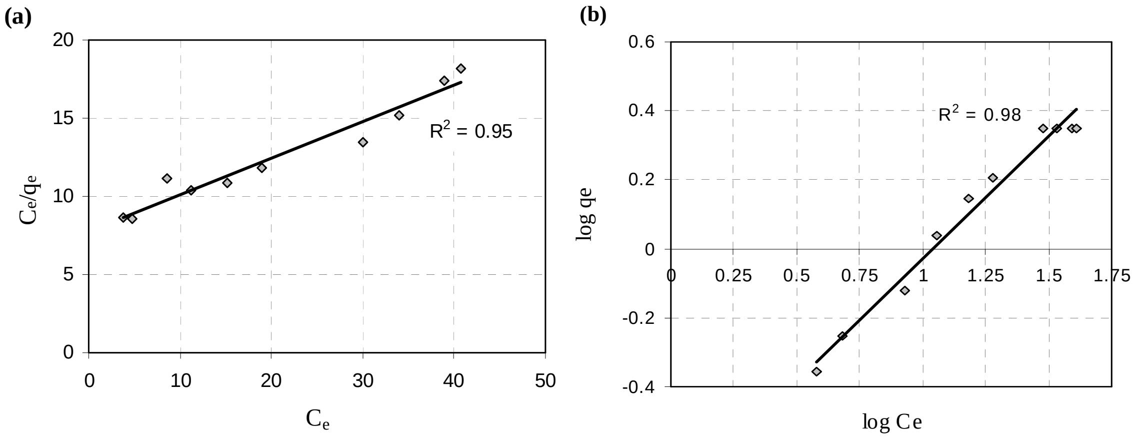


***Figure 4.*** *Isothermal curves of bare palm adsorption (1.0 mm particle size, 30 °C) (a) Langmuir (b) Freundlich*

### *3.3. Adsorption Kinetics*

The study of the adsorption kinetics describes the resistance to solute transfer from the solution to the boundary layer at the solid-liquid interface to the pore water and then to the solid.

It is well known that adsorption kinetics is mainly controlled by the following steps:

1. Solute molecules transfer from the solution to the boundary film;
2. Solute molecules transfer from the film to the surface of the sorbent (external diffusion);
3. Diffusion from the surface to intra-particle sites and
4. Interaction of solute molecules with the available sites on the internal surface [29]

***Table 1.*** *Comparison of Langmuir and Freundlich constants for the dye adsorption on bare palm branches*

| Adsorption condition (mm particle size, °C) | Langmuir | | | | Freundlich | | |
|---|---|---|---|---|---|---|---|
| | $K_L$, ($dm^3$/g) | $a_L$, ($dm^3$/mg) | $q_{max}$, (mg/g) | $R^2$ | $K_F$, ($dm^3$/g) | n | $R^2$ |
| (2.0 mm particle size, 30 °C) | 0.041 | 0.0097 | 4.17 | 0.49 | 0.023 | 0.79 | 0.97 |
| (1.2 mm particle size, 30 °C) | 0.735 | 0.0051 | 14.30 | 0.13 | 0.069 | 1.11 | 0.95 |
| (1.0 mm particle size, 30 °C) | 0.129 | 0.0303 | 4.277 | 0.94 | 0.182 | 1.41 | 0.98 |
| (1.0 mm particle size, 40 °C) | 0.137 | 0.3282 | 4.185 | 0.95 | 0.193 | 1.43 | 0.98 |
| (1.0 mm particle size, 50 °C) | 0.146 | 0.0372 | 3.918 | 0.86 | 0.192 | 1.42 | 0.86 |
| (1.0 mm particle size, 60 °C) | 0.167 | 0.0433 | 3.849 | 0.89 | 0.273 | 1.65 | 0.82 |

$R^2$: Correlation factor

***Table 2.*** *Comparison of first and second order reaction kinetic constants for the dye adsorption on the bare palm branches*

| Adsorption condition | | | Pseudo-first order kinetic model | | Pseudo-second order kinetic model | |
|---|---|---|---|---|---|---|
| Air flow rate ($cm^3$/min) | Initial concentration Of the dye (mg/$dm^3$) | Mass adsorbent (g) | $K_1$ (*$10^2$), ($min^{-1}$) | $R^2$ | $K_2$ (*$10^3$), (g.$mg^{-1}min^{-1}$) | $R^2$ |
| 20 | 376.8 | 5.0 | -0.008 | 0.3 | 0.0066 | 0.001 |
| 40 | 376.8 | 5.0 | -0.009 | 00.6 | 0.0076 | 0.99 |
| 60 | 376.8 | 5.0 | -0.008 | 0.6 | 0.0076 | 0.99 |
| 80 | 376.8 | 5.0 | -0.005 | 0.3 | 0.0012 | 0.99 |
| 80 | 97.8 | 5.0 | -0.008 | 0.2 | 0.0200 | 0.99 |
| 80 | 198.7 | 5.0 | -0.020 | 0.8 | 0.0039 | 0.99 |
| 80 | 296.5 | 5.0 | -0.008 | 0.5 | 0.0025 | 0.99 |
| 80 | 376.8 | 5.0 | -0.01 | 0.3 | 0.0120 | 0.99 |
| 80 | 376.8 | 1.0 | -0.008 | 0.6 | 0.0050 | 0.99 |
| 80 | 376.8 | 2.0 | -0.009 | 0.9 | 0.0080 | 0.99 |
| 80 | 376.8 | 3.0 | -0.009 | 0.6 | 0.0060 | 0.99 |
| 80 | 376.8 | 5.0 | -0.008 | 0.3 | 0.0012 | 0.99 |

$R^2$: Correlation factor; $K_1$ and $K_2$ are first and second reaction kinetics, respectively

The evolution of the adsorption process can be followed by measuring the number of particles adsorbed per unit time. Many kinetic models have been proposed for the adsorption of solutes on solids. They include, amongst others, the pseudo-first order kinetic model and the pseudo-second order model. Examination of the results presented in Table 2 shows that the adsorption data fitted best to the pseudo-second order rate equation since the correlation coefficient is close to one and higher than that for the first order.

Thus, this applicable to describe the adsorption process of the dye onto bare palm, based on assumption that the rate-limiting step maybe chemical sorption involving valency forces through sharing or exchange of electrons between sorbent and sorbate, provide the best correlation of data [10]. Similar results were reported for the adsorption of oil by powder and flake chitosan Ahmad et al., [31].

## 4. Conclusions

The present study demonstrated that bare palm branches, which are abundantly available in Egypt, is an efficient sorbent for the removal of dye in water and it may be an alternative to more costly adsorbents such as activated carbon. The batch studies clearly suggest that bare palm sorbent exhibits almost 100% adsorption at lower concentrations of the dye. Equilibrium data fitted well with the Freundlich model, which suggests a heterogeneous coverage of dye molecules on the surface of the bare palm branches. Moreover, the kinetic data were best fitted to pseudo-second order kinetic model.

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