

Potential Application of Propionic Acid Modified Sugarcane Bagasse for Removing of Basic and Acid Dyes from Industrial Wastewater

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Abstract Raw sugarcane bagasse modified with propionic acid (RBMPA) was used as an efficient, low cost and eco-friendly adsorbent for the removal of methylene blue (basic) and orange II (acid) dyes from aqueous solutions. The effect of different system variables, namely contact time, dye concentration, adsorption dose, pH and particle size were investigated and optimal experimental conditions were ascertained. A greater percentage of dye removal was observed in acidic medium (pH=2) in case of orange II whereas in (pH=7) in case of methylene blue dyes and increases with the increase of the dosage of adsorbent. Maximum dye removal was attained after 60 min. Equilibrium isotherms were analysed by the Langmuir and Freundlich models of adsorption and it was found that the Langmuir isotherm provides a good model for the adsorption of methylene blue whereas the Freundlich isotherm is fit for the adsorption of orange II. The kinetics of the adsorption of both dyes was consistent with pseudo second-order kinetics with a good correlation ($R^2 > 0.99$). Furthermore, the amounts of dye adsorbed at equilibrium, q_e (mg g⁻¹) for methylene blue and orange II were 59.5 and 25.5, respectively.

Keywords Modified Bagasse, Adsorption, Methylene Blue, Orange II, Kinetics

1. Introduction

Dyes are type of organic compounds that can provide bright and lasting color to other substances¹. There are more than 100,000 dyes available commercially, which are specifically designed to resist fading upon exposure to sweat, light, water, and oxidizing agents and, as such, are very stable and difficult to degrade². Synthetic dyes have been increasingly used in the textile, leather, paper, rubber, plastic, cosmetics, pharmaceuticals, and food industries. Contamination of water sources by visible colored pollutants mainly dye stuff has become a major problem for the environment. The colored wastewater discharged into environmental bodies of water is not only an esthetically unpleasant but also interferes with light penetration and reduce photosynthetic action. Many dyes or their metabolites have toxic as well as carcinogenic, mutagenic, and teratogenic effects on aquatic life and humans³. Hence, the removal of dyes from wastewater is essential to prevent continuous environmental pollution.

The conventional wastewater treatment, which rely on

aerobic biodegrading have low removal efficiency for reactive and other anionic soluble dyes. Due to low biodegrading of dyes, a conventional biological treatment process is not very effective in removing of dyes from wastewater. It is usually treated either by physical or chemical processes. However, these processes are very expensive and cannot effectively be used to treat the wide range of dyes waste⁴. The adsorption process is one of the effective methods for removal dyes from the waste effluent.

Activated carbon (powder or granular) is the most widely used adsorbent because it has an excellent adsorption efficiency for organic compounds. But, commercially available activated carbon is very expensive. This had lead to further studies for cheaper substitutions. Nowadays, there are numerous numbers of low cost, commercially available adsorbents which had been used for the dye removal. A variety of agricultural biosorbents, including bagasse, have been recently investigated for their dye removal ability⁵⁻⁸. The chemical modification of sorbents with acids or bases was reported in the literature in order to enhance their adsorption capacity^{3, 9}.

The removal of methyl red from simulated wastewater by using modified bagasse has been investigated for different variables¹⁰. They have shown that sugarcane bagasse treated with phosphoric acid and untreated sugarcane ba-

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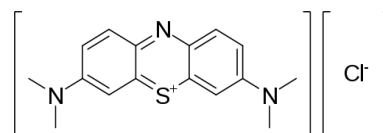
gasse has a lower adsorption efficiency compared to powdered activated carbon. Congo Red adsorption by ball-milled sugarcane bagasse has been studied by Zhang *et al.*¹¹. They found that the adsorption capacity of Congo Red increased significantly with small changes in bagasse surface area. Maximum capacity was 38.2 mg g^{-1} at a concentration of 500 mg L^{-1} . Karla *et al.*¹² studied the application of succinylated sugarcane bagasse as adsorbent to remove methylene blue and gentian violet from aqueous solutions. The results revealed that, the adsorption processes could be described by pseudo-second order kinetic model. Adsorption isotherms were well fitted by Langmuir equation. Moreover, the maximum capacities for methylene blue and gentian violet were found to be 478 and 1273.2 mg g^{-1} . Sugarcane bagasse modified with tetraethylenepentamine for removal of eosin Y has been studied by Gang-Biao *et al.*¹³. They concluded that the modified bagasse exhibited interesting sorption properties towards anionic dyes to the plentiful introduced amino groups. The experimental data showed also that the sorption process was rapid and followed a pseudo-second order model and well fitted Langmuir's isotherm model. However, the use of modified sugarcane bagasse with propionic acid for removing methylene blue and orange II dyes from wastewater to our knowledge has not yet reported. Therefore, in this paper, we studied the performance of sugarcane bagasse modified with propionic acid on the removal of the basic dye methylene blue and the acid dye orange II. We also studied several factors influencing the adsorption efficiency such as pH, contact time, dye concentration, biomass dosage and particle size. The equilibrium adsorption capacity of both dyes on the modified bagasse was determined making use of the Freundlich and Langmuir isotherm equations. Furthermore, the adsorption kinetics of the two dyes was studied by application of pseudo-first and second-order models.

2. Materials and Methods

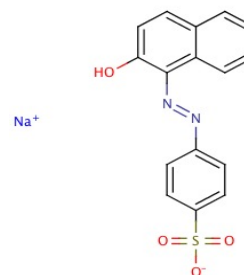
2.1. Preparation of Adsorbent

Sugarcane bagasse was purchased from the Egyptian sugar and integrated industries company (ESIIC) and were subjected to repeatedly washing with distilled water to remove dust and soluble impurities. It was dried overnight at 60°C before crumbing and shredding. The dried and shredded bagasse was then stored in a glass container and designated as a raw bagasse (RB). The propionic acid modified RB was prepared by mixing 5 wt. % acid with RB at room temperature. Finally the treated RB was dried in an oven at 60°C overnight and stored and labelled as raw sugarcane bagasse modified with propionic acid (RBMPA). A stock solution of dyes (600 and 300 mg L^{-1}) of methylene blue and orange II respectively were prepared and stored in a dark place and used freshly. All the chemicals used throughout this study were of analytical grade reagents. The chemical structures of the two dyes are presented in Fig.1.

Double distilled water was used for preparing all of the reagents solutions throughout the study. All the adsorption experiments were carried out at room temperature ($30 \pm 2^\circ\text{C}$) using 1 gm (0.25 mm) and pH's 7.0 and 2.0 for the adsorption of methylene blue and orange II respectively.



(a) Methylene blue



(b) Orange II

Figure 1. The chemical structure of methylene blue (a) and orange II (b)

2.2. Methods

All pH measurements were carried out with a pH-meter model number JENWAY 3450. The initial pH values were adjusted with 0.1 M HCl or NaOH to form a series of pH 's from 2 to 11. Concentration of dye solutions were estimated using a thermo Fischer scientific, aquamate, and evolution 166 UV-visible spectrophotometer.

2.3. Adsorption Study

The adsorption study was mainly carried out by batch technique to obtain rate and equilibrium data. The experiments were performed to observe the effect of pH, contact time, particle size, concentration, etc. One gram of adsorbent was taken in 100 mL airtight volumetric flasks containing 50 mL each of different adsorbate solutions of known concentrations. The flasks were then shaken intermittently for 1 hr to attain the equilibrium condition of adsorption at room temperature. The solutions obtained were then filtered using Whatmann filter paper and analysed spectrophotometrically by measuring the absorbance at λ_{max} of each dye.

2.4. Kinetic Studies

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

reported. The maximum deviation observed was less than $\pm 5\%$.

3. Results and Discussion

3.1. Effect of Contact Time

The contact time between pollutant and the adsorbent is of significant importance in the wastewater treatment by adsorption. A rapid uptake of pollutants and establishment of equilibrium in short period signifies the efficiency of that adsorbate for its use in wastewater treatment. Available adsorption studies in literature reveal that the uptake of the adsorbate species is fast at the initial stages of the contact period, and therefore, it becomes slower near the equilibrium. The effect of contact time for the adsorption of methylene blue and orange II was studied for a period of 240 min and the results are shown in Fig.(2). It shows that the dye removal was rapid in the first 10 min and the curves of contact time are single, smooth and continuous leading to saturation. These curves indicate the possible mono-layer coverage of dye on the surface of modified bagasse¹⁴⁻¹⁶. In addition the adsorption of methylene blue is faster than that of orange II and the equilibrium was attained after 60 min. The percentage removal of methylene blue dye after 2 min was found equal to 84% relative to 62% for orange II dye. Furthermore, these results reveal that the available sites on the RBMPA surface are more active towards adsorption of methylene blue than the adsorption of orange II.

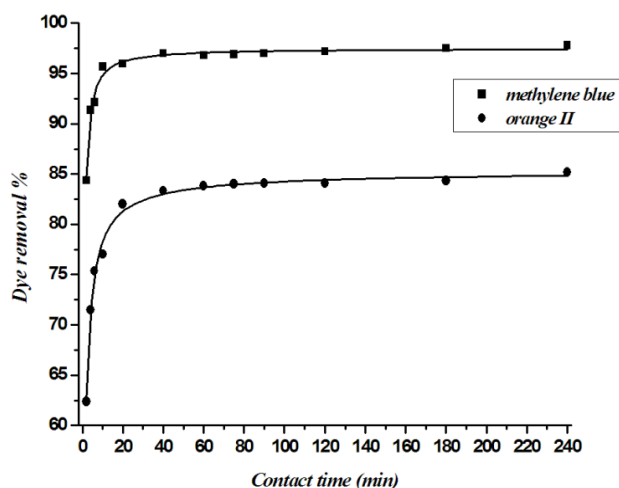


Figure 2. Variation of dye removal with contact time for the adsorption of methylene blue and orange II onto RBMPA

3.2. Effect of Initial Dye Concentration

The effect of initial methylene blue and orange II concentrations for their removal by RBMPA is shown in Fig (3&4), from these figures it is evident that as the initial dye concentration increased, the adsorption capacity increased which exhibited a strong linear relationship for both dyes with the correlation coefficient (R^2) close to unity. However, the adsorption capacity of RBMPA to methylene blue reached to 100 % when the dye concentration increased

from 25 up to 450 mg L^{-1} . On the other hand, the adsorption capacity of RBMPA to the orange II attained 100 % when the dye concentration increased from 25 up to 100 mg L^{-1} . These results revealed that the available active sites on RBMPA surface have greater tendency to the adsorption of methylene blue than that of orange II. These results may indicate that there is a possibility to treat textile effluent with relatively higher dye concentration using low cost RBMPA.

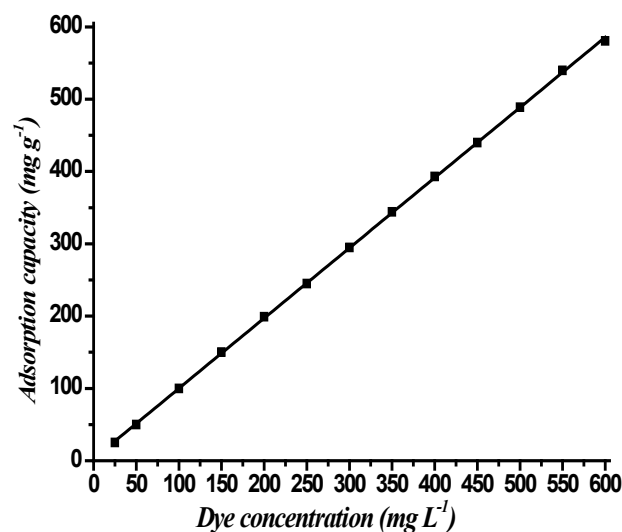


Figure 3. Effect of methylene blue concentration with the adsorption capacity by RBMPA at pH=3.0

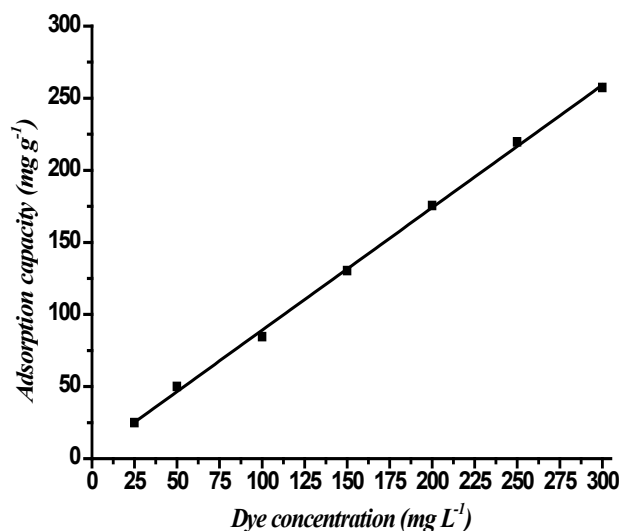


Figure 4. Variation of orange II concentration with the adsorption capacity by RBMPA at pH=2.0

3.3. Effect of pH

The pH of the solution affects the surface charge of the adsorbents as well as the degree of ionization of different pollutants. Change of pH also affects the adsorptive process through dissociation of functional groups on the adsorbent surface active sites. Consequently, this leads to a shift in reaction kinetics and adsorption equilibrium. The adsorption of methylene blue and orange II over a pH range of 2-11 were carried. The results presented in Fig. 5 indicate

that the % dye removal for methylene blue was 90 % and increased with the increase of pH up to 3.0 keeping its value (97%) on increasing the pH value up to 11. On the other hand the maximum % dye removal (80 %) for orange II was found at pH=2 and followed by a contentious decrease on increasing the pH up to 4 with value of 38 %. Further increase in the pH value is accompanied with no change in the dye removal. The high percentage removal may be attributed to a significantly high electrostatic attraction exists between the positively charged surface of the adsorbent and the anionic dye¹⁷. It is interest to mention here that as the pH of the system increased above 3 the dye removal of methylene blue is kept constant indicating the number of the positively charged sites is unchanged. On the other hand, on increasing the pH above 2 a negatively charged surface sites on the adsorbent did not favour the adsorption of orange II dye anions due to electrostatic repulsion. In conclusion the removal of methylene blue is unaffected by increasing the pH value, whereas the removal of orange II is drastically affected by increasing the pH value.

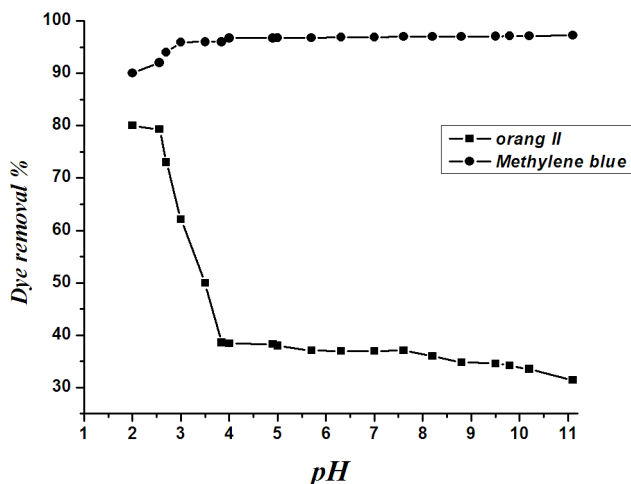


Figure 5. Effect of pH on adsorption of methylene blue and orange II onto RBMPA

3.4. Effect of Adsorbent Dosage

The effect of RBMPA dosage on the removal of methylene blue and orange II are shown in Fig. (6). the percentage removal increased with the RBMPA dosage up to a certain limit then it reached a constant value. The increase in adsorption of dyes with adsorbent dosage was due to the availability of more adsorption sites on bagasse surface for adsorption. The results clear that remarkable removal of the methylene blue dye in comparison with the % removal of orange II in presence of 0.2 g adsorbent. This behaviour indicates that the number of available sites on the RBMPA surface is active towards the adsorption of methylene blue.

3.5. Effect of Particle Size

The particle size has a significant influence on the kinetic of adsorption. The influence of particle size furnishes important information for achieving optimum utilization of adsorbent. Rate of adsorption on solid surface is expected to

vary with available surface area for a constant adsorbent weight with the particle size.

The adsorption capacity is directly proportional to the total exposed surface and inversely to the particle diameter for non-porous adsorbents. The presence of a large number of smaller particles provides the adsorption system with a greater surface area available for dye removal¹⁸. Fig. 7 shows the results of removal of methylene blue and orange II dyes on the different particle sizes (0.25-1.0 mm). It was observed that as the particle size decreased the amount of adsorbed dye increased. This is due to the large external surface area available from fine particles¹⁹. Furthermore, the variation of methylene blue removal is higher than that of orange II and quite constant with the different particle size. This behaviour means that the available active sites on RBMPA are little changed with the different particle size.

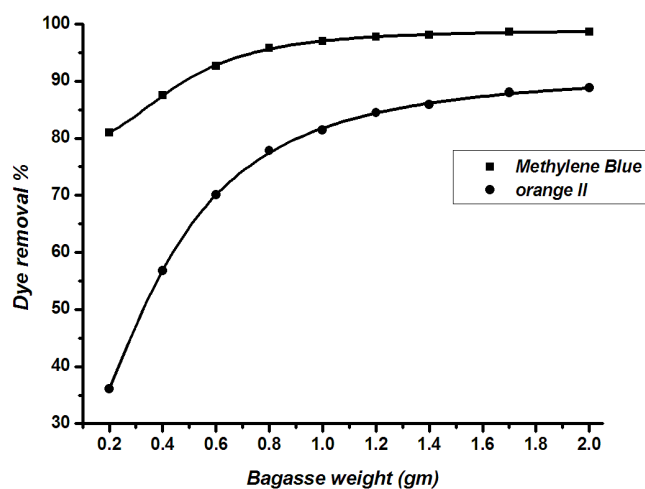


Figure 6. Effect of RBMPA dosage on the adsorption of methylene blue and orange II

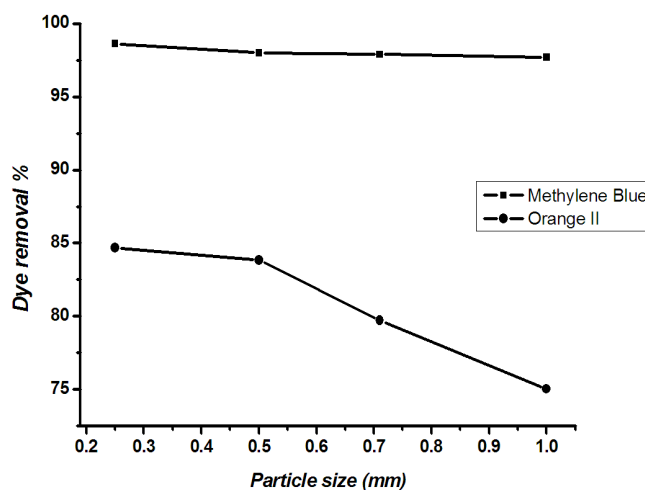


Figure 7. Effect of particle size of RBMPA on the adsorption of methylene blue and orange II

3.6. Adsorption Equilibrium Study

Equilibrium isotherm equations are used to describe the experimental adsorption data. The parameters obtained from the different models provide important information on

the adsorption mechanisms and surface properties and affinities of the adsorbent. The most accepted surface adsorption models for single-state systems are the Langmuir and Freundlich models.

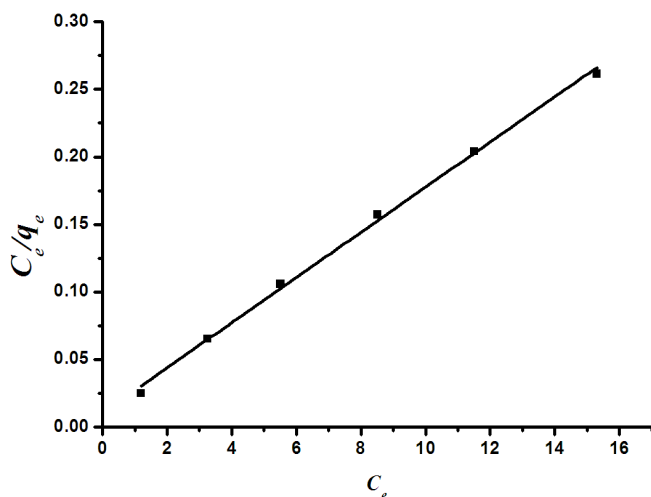


Figure 8a. Langmuir plot for the removal of methylene blue dye by RBMPA

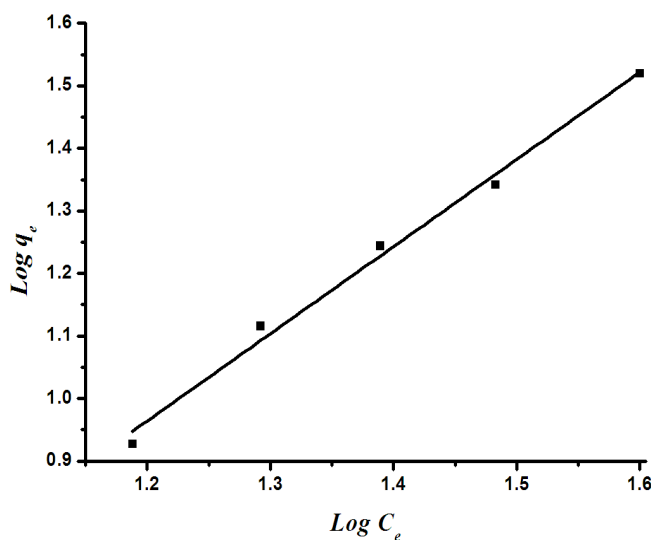


Figure 8b. Freundlich isotherm plot for the removal of orange II dye by RBMPA

Langmuir isotherm

A basic assumption of the Langmuir theory is that adsorption takes place at specific homogenous sites within the adsorbent. It is then assumed that once a dye molecule occupies a site, no further adsorption can take place at that site. Theoretically, therefore, a saturation value is reached, i.e., monolayer adsorption. The Langmuir isotherm is represented by the following equation:

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

where q_e (mg g^{-1}) is the amount of dye adsorbed at equilibrium, C_e (mg L^{-1}) is the equilibrium concentration of the dye, Q_m is a constant and reflects a complete monolayer (mg g^{-1}) and K_a is the adsorption equilibrium constant (Lmg^{-1}) that is related to the apparent of adsorption.

The Langmuir isotherm (Eq.1) can be linearised into the

following form:

$$C_e/q_e = 1 / (K_a Q_m) + C_e / Q_m \quad (2)$$

The Q_m and K_a values can be obtained from the slope ($1/Q_m$) and intercept ($1/K_a Q_m$) of the linear plot of C_e/q_e versus C_e .

Freundlich isotherm

The Freundlich isotherm is derived by assuming a heterogeneous surface with a non-uniform distribution of heat of adsorption over the surface. The Freundlich isotherm is represented by the following equation:

$$q_e = K_F C_e^{1/n} \quad (3)$$

Where q_e and C_e are defined as before in eq. (1) and K_F is a constant related to bonding energy and adsorption capacity. $1/n$ indicates the adsorption intensity of dye onto the adsorbent and the type of isotherm to be irreversible ($1/n=0$), favourable ($0<1/n<1$) and unfavourable ($1/n>1$).

This equation can be linearized as follow:

$$\text{Log } q_e = \text{Log } K_F + 1/n \text{ Log } C_e \quad (4)$$

Fig.8 (a&b) illustrates the Langmuir and Freundlich linear isotherm for methylene blue and orange II. The coefficients isotherm parameters for both dyes on RBMPA are shown in Table (1). The correlation coefficients reported in Table (1) show strong positive evidence on the adsorption of the two dyes on modified bagasse. The applicability of the linear form of Langmuir and Freundlich models to modified bagasse was proved by the high correlation coefficients $R^2>0.99$. This suggests that the Langmuir isotherm provides a good model for the adsorption of methylene blue whereas the Freundlich isotherm is a suitable model for the adsorption of orange II.

Table 1. Langmuir and Freundlich parameters for the removal of methylene blue and orange II dyes by RBMPA

| dye | Freundlich | | |
|----------------|------------|--------------------------|------------------------|
| | R^2 | K_F | n |
| Methylene blue | - | - | - |
| Orange II | 0.9902 | 0.1959 | 0.7176 |
| dye | Langmuir | | |
| | R^2 | $K_a(\text{Lmg g}^{-1})$ | $Q_m(\text{mgg}^{-1})$ |
| Methylene blue | 0.9974 | 1.5755 | 59.8802 |
| Orange II | - | - | - |

3.7. Adsorption Kinetic Study

In order to investigate the adsorption process of methylene blue and orange II dyes on RBMPA pseudo-first-order and pseudo-second-order were used. Pseudo-first-order and pseudo-second-order rate equations are expressed as follows:

$$\text{Log } (q_e - q_t) = \text{Log } q_e - K_1 t / 2.303 \quad \text{pseudo-first-order} \quad (5)$$

$$t/q_t = 1/h + t/q_e \quad \text{pseudo-second-order} \quad (6)$$

where q_e and q_t are the amount of adsorbed dyes (mg g^{-1}) at equilibrium and at time t , respectively, K_1 is the rate constant of pseudo-first-order adsorption (min^{-1}), h ($K_2 q_e^2$) is the initial adsorption rate ($\text{mg g}^{-1} \text{min}^{-1}$) and K_2 is the rate constant of pseudo-second-order adsorption ($\text{mg g}^{-1} \text{min}^{-1}$). The straight lines obtained and presented in Fig. 9 indicate the applicability of the above equations. The values of K_1

and K_2 were calculated from the slopes of the respective linear plots and are noted in **Table (2)**. Comparing the correlation coefficients of both equations it was to be concluded that the kinetic model for methylene blue and orange II is consistent with a pseudo-second-order ($R^2 > 0.99$).

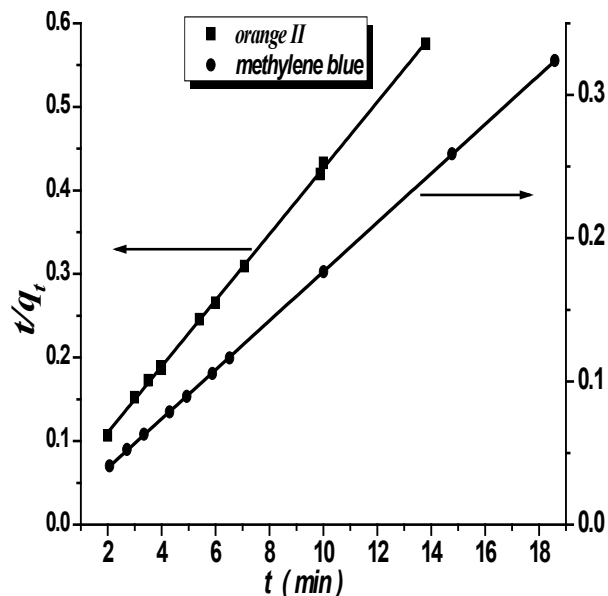


Figure 9. The pseudo-second-order plot for methylene blue and orange II dyes

Table 2. the pseudo-second-order parameters for methylene blue and orange II dyes

| Dye | R^2 | K_2 ($\text{mg g}^{-1}\text{min}^{-1}$) |
|----------------|--------|---|
| Methylene blue | 0.9999 | 0.0548 |
| Orange II | 0.9995 | 0.0501 |

3.8. Intra-particle Diffusion Study

Weber-Morris²⁰ found an empirical function relationship common to most adsorption processes in which the uptake varies almost proportionally with $t^{1/2}$ as the following equation:

$$q_t = K_{id} t^{1/2} + C \quad (7)$$

Where K_{id} is the intra-particle diffusion rate constant, q_t (mg g^{-1}) is the amount of dye adsorbed at time t (min). A plot of q_t versus $t^{1/2}$ should be a straight line with a slope K_{id} and intercept C . Values of intercept give an idea about the thickness of the boundary layer, i.e., the larger the intercept the greater the boundary layer effect²¹. Fig. 10 shows plots of mass of dyes adsorbed q_t versus $t^{1/2}$ for methylene blue and orange II. The results indicate that the adsorption process of both dyes exhibits two separate regions with two straight lines. The values of intercept, slope and the correlation coefficient (R^2) were calculated and presented in Table 3. The deviation of the straight lines from the origin (Fig. 10) may be due to the difference in the rate of mass transfer in the initial and final stages of adsorption²². Further, the first straight portion is attributed to a macropores diffusion process and the second linear portion can be ascribed to a micropore diffusion process²³. In addition, it is clear from Fig. 10 that the first stage is faster than the second one. This

behaviour may be correlated with the very slow diffusion of the adsorbate from the surface film into the micropores, which are the least accessible sites for adsorption. Furthermore, the values of R^2 clearly prove the good applicability of the Weber-Morris equation²⁰. The parameters of the intra-particle diffusion data for methylene blue and orange II are listed in **Table 3**.

Table 3. Intra-particle diffusion model parameters for methylene blue and orange II dyes

| dye | Stage I | | |
|----------------|----------|--------|-----------|
| | R^2 | Slope | Intercept |
| Methylene blue | 0.9915 | 5.9335 | 42.0435 |
| Orange II | 0.9833 | 3.5484 | 13.6684 |
| dye | Stage II | | |
| | R^2 | Slope | |
| Methylene blue | 0.9687 | 0.7354 | |
| Orange II | 0.9499 | 0.4486 | |

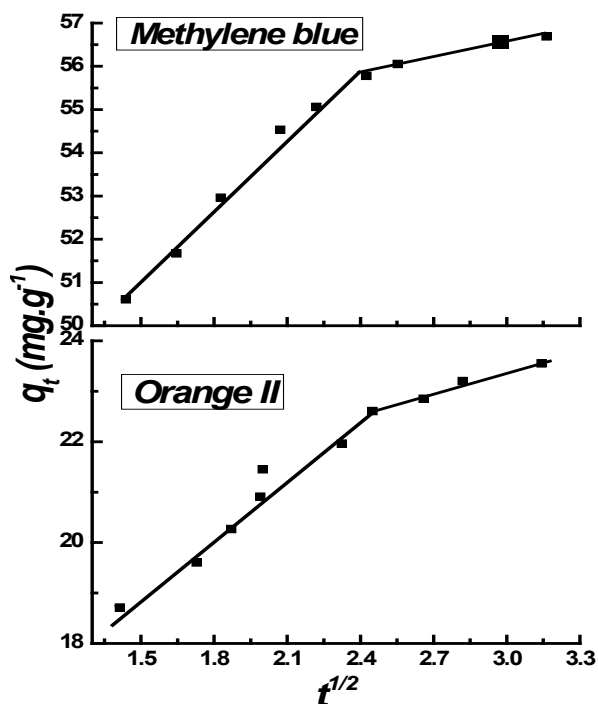


Figure 10. Intra-particle diffusion model for methylene blue and orange II dyes

4. Conclusions

The present study shows that sugarcane bagasse modified with propionic acid (RBMPA) is an effective adsorbent for the removal of methylene blue and orange II dyes from aqueous solutions. The effective pH for methylene blue and orange II were 7 and 2.0 respectively. Quasi-equilibrium was practically achieved in 60 min. The adsorption equilibrium data of the two dyes indicated that the adsorption of methylene blue well fitted the Langmuir equation whereas the Freundlich equation is an applicable model for the adsorption of orange II. Adsorption kinetics revealed that methyl-

ene blue and orange II follow a pseudo-second-order model. The intra-particle diffusion results pointed to the presence of macropores in the first stage and micropores as a second slow stage of the adsorption process. The present study concluded that the RBMPA could be employed as a low cost and ecofriendly adsorbent as an alternative to commercial activated carbon for the removal of dyes from wastewater.

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