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Original Article

Synthesis and Characterization of Activated Carbon from Rice Husk for Wastewater Treatment

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Abstract

In this study, activated carbon (AC) was utilized for removal of methylene blue dye (MB) from wastewater. The activated carbon was prepared from rice husk through thermal and chemical activations. The morphology and size of prepared activated carbon were investigated by transmission electron microscope (TEM). The batch adsorption technique was used to study the removal of methylene blue form wastewater. Different parameters such as adsorbent dosage (0.1-0.6 g/100 mL), initial pH (3-10), initial dye concentration (5-150 mg/L) and contact time (1-60 min) were examined. The results revealed that the reaction proceeded by pseudo–second order model and the Langmuir isotherm model was the predominant mechanism with a maximum adsorption.

1. Introduction

Recently, there is a growing concern on pollution study; since it can affect the plant, animal and human beings. The quick progress in industry is an excellent fact but at the same time can cause a severe threat to all living organisms and disturb the ecological system. Water is one of the backbones of the environment and its contamination leads to many problems for all living organisms **(Iqbal et al., 2019)**. The most common contaminants for water are nutrients (nitrogen and phosphorus), heavy metals, hydrocarbons, organic matter and microbes **(Akpor et al., 2014).**

The dye production represents an interesting sector in the chemical industries. The production of dyes all over the world is nearly 800,000 tons per year all over the world. The synthetic dyes are applied in various industries such as paper printing, textile, pharmaceutical, food, cosmetics, and leather. Throughout the various processes of textile industry, about 10-15% of dyes are missing **(Hassaan and El Nemr, 2017).** This leads to production of a huge quantity of wastewater in dyeing and printing process which contains large amount of coloring agents. It is well known that the aquatic organisms require light to create energy while the presence of color from days can prevent the light penetration leading to discrepancy of ecosystem **(Iqbal et al., 2015).**

All dyes are water-soluble and hold traces of metals such as Cr, Co, Cu, Zn and Pb in their aqueous solution except vat and disperse dyes**.** Dyes are broadly classified into cationic, anionic, and non-ionic. Anionic dyes comprise different dyes groups such as acid, reactive azo and direct dyes while cationic dyes are the basic ones **(Dawood and Sen, 2014).**

The greatest significant basic dye is methylene blue, discovered in 1878 by Caro. It is a dark-green powder or crystalline solid. It is widely used as a stain and has some biological uses, but it has different unsafe effects on the human beings, so it should be eliminated from wastewater. It dissociates in aqueous solution into methylene blue cation and the chloride ion. The colored cation can be adsorbed by several adsorbents **(El Qada et al., 2006).** For wastewater treatment, chemical and physical methods such as coagulation-flocculation, precipitation, advanced oxidation, ion exchange, membrane filtration, etc. have been reported.

Agricultural wastes have been broadly explored to produce inexpensive adsorbents for eliminating of various types of pollutants (heavy metals ions, hydrocarbons and dyes) from water and wastewater **(Besharati et al., 2018).** For example; rice husk is a one of the inexpensive biomass in which the constituents like cellulose (55%-60%), hemicellulose (20%-25%) and lignin (20%-25%) are considered as promising precursors for preparation of activated carbon (AC) along with some functional groups such as carboxyl, hydroxyl and amidogen, etc., which make rice husk as a potential adsorbent material **(Sugashini and Begum., 2015).**

AC is broadly used for the elimination of dyes from aqueous solutions because it has high adsorption capacity, large surface area and chemical affinity for ionic and non-polar species. The only limitation for using AC in wastewater treatment is the high price of the commercially available AC. However, biochar that possesses similar AC properties can be obtained from agricultural waste materials with little economic value and can be used as alternative carbon adsorbent material **(Andreou and Pashalidis, 2018).**

Adsorption process is a promising method for treatment of dye contaminated effluents compared with different dye removal technologies due to its low cost, sludge-free process, produce low amounts of waste and enable dye recovery **(Andreou and Pashalidis., 2018).**

In the present study, an attempt to synthesize AC from rice husk was conducted. The prepared AC was utilized as adsorbent for removal of methylene blue dye form wastewater. Also, the operational factors such as pH of solution, contact time, adsorbent dose, dye concentrations were studied. Moreover, the equilibrium data and kinetic parameters were evaluated by adsorption isotherm model.

2. Materials and Methods

Rice husk starting material was obtained from Sharkia governorate, Egypt. The rice husk was washed with water to remove the dirt and other contaminants, dried in air for 24 h then ground and sieved to small fractions to be ready for AC production. On the other hand, methylene blue (cationic dye), hydrochloric and phosphoric acids were purchased from Sigma Aldrich.

2.1. Preparation of AC from rice husk

200 g of prepared rice husk was firstly carbonized at 200 and 400 °C for 1 hour in a muffle furnace in presence of argon atmosphere to obtain rice husk ash. Then, 100 g of the prepared rice husk was subjected impregnation in 200 ml of 3 molar H_3PO_4 at 80 °C for 3 h. After that, the chemically activated ash was cleaned with water until its pH value was 7. The sample was dried at 100 °C for 24 hr. Then, the dried sample was carbonized at 450 and 700 °C for 2 hours in a muffle furnace under argon. Finally, the activated carbon was crushed and sieved to obtain fine activated carbon **(Cheenmatchaya and Kungwankunakorn, 2014).**

2.3. Characterization of prepared AC

The morphology and size of the prepared AC adsorbent were investigated by transmission electron microscope (TEM). TEM offers valuable information on the inner structure of the materials such as crystal structure, morphology, stress-state information, and size of the particle. TEM type Philips, EM, 208 was utilized to examine the prepared adsorbents.

2.4. Adsorption study

In the present study, the experimental work was carried out by batch adsorption process. A stock solution of MB having concentration of 1000 mg/l was prepared. For the experimental study, 100 ml aqueous solution of MB with varying known concentrations were taken in 250 ml conical flask containing a known amount of adsorbent. These flasks were placed in a thermostatic shaker at 120 rpm at a temperature of 25°C. The effect of various parameters such as contact time, adsorbent dose (50-350 mg/100 ml), pH (3-10) and concentration of dye (5–150 mg/l) were studied.

The adsorbent was separated by centrifugation or filtration. The residual concentration of MB was determined by spectrophotometer type Perkin Elmer lambda 25, at 665 nm. The percentage of dye removal from solution was calculated by the following equation:

$$\textbf{Removal \%} = \frac{C0-Ct}{C0} * 100$$

where C_0 and C_t (mg/l) are the initial dye concentration and concentration at time t, respectively.

3. Results and discussion

3.1. Morphology and size of the prepared AC

Figure 1 shows TEM images of the prepared AC. It is indicated that the AC comprises of homogeneous particle morphology and particle size. The images include spherical, elongated and gel-like particles; on the other hand, the particles exhibit wide range of sizes (10-300nm). According to the original constituents of rice husk, it is possible to find fine silica particles in the TEM images. This means that the prepared AC should include some silica particles.

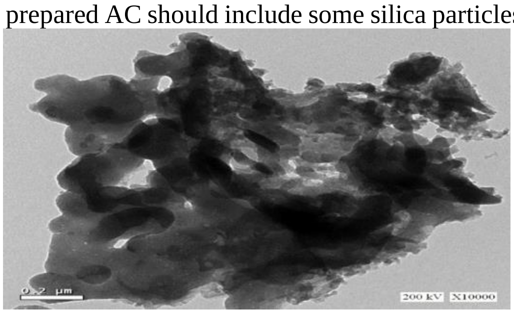

Figure 1: TEM images of prepared AC

3.2 Adsorption study of activated carbon

3.2.1. Optimization of the MB dye removal by AC

3.2.1.1 Effect of pH:

Solution pH is an important parameter that affects the surface charge of the adsorbent, the ionization extent as well as the structure and specificity of adsorbate

(Ranga, and Sanghvi., 2015). In order to examine the effect of pH on the adsorption of MB dye onto AC, batch equilibrium studies were carried out at pH range (3–10); the results are shown in Figure (2). It is obvious that increasing pH leads to increase in the removal percentage of MB dye until pH 9. At lower pH values, i.e. in the acidic range, the surface tends to acquire a net positive surface charge due to the presence of H_3O^+ ions which causes a decrease in the adsorption of MB (cationic dye) that faces repulsion from the positively charged surface and competition with H_3O^+ ions. With increasing pH, the cationic MB dye is attracted to the negatively charged AC surface where functional groups as hydroxyl and carboxyl predominate (**Singh and Dawa, 2014).**

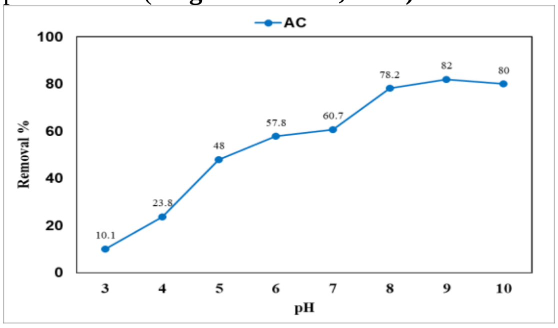

Figure 2: Effect of pH on removal of MB by AC (50 mg /L MB, adsorbent dose = 0.1 g/ 100 ml, Temp. = 25◦C, and contact time = 120 min.)

3.2.1.2. Effect of adsorbent dose:

The influence of AC dosage (0.1-0.6 gm) on the removal percentage of MB dye (50 ppm) at 25°C was investigated. The data presented in Figure (3) exhibit that with increasing the adsorbent dose from 0.10 g to 0.60 g, the removal percentage of MB increases from 33% to 83%. This is due to the increase of available surface area of active centres. However, with increasing adsorbent dose above 5 g there is no significant increase in dye removal percentage. This can be interpreted as the available number of active sites is increased with higher doses; this might hinder the adsorption process and less number of dye molecules can occupy the active site **(Geçgel et al., 2012; Singh et al., 2019).**

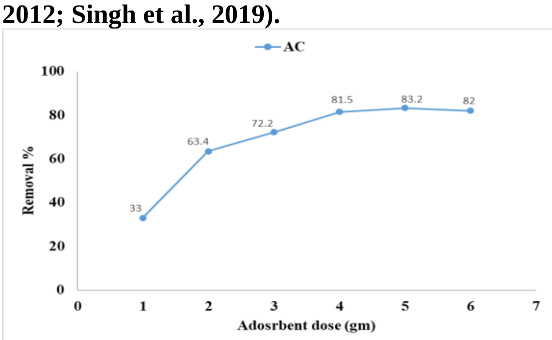

Figure 3: Effect of adsorbent dosage on MB removal by AC (50 mg/ l MB, pH = 7.5, contact time = 120 min, at 25◦C)

3.2.1.3. Effect of contact time

The effect of agitation time (0-120 min.) on the removal percentage of MB dye by AC is displayed (Figure 4). It can be observed that the percentage of dye removal is increased from 50.3% after 3 min to approximately 80% after 60 min. The adsorption is started rapid initially and then gradually increased until the equilibrium is reached. This can be interpreted by the fact that a large number of vacant sites are available initially at the surface of the adsorbent which gradually decrease with time as most of the sites are occupied within 60 min (**Singh and Dawa, 2014).** Thus, the contact time 60 min is chosen as the optimum time in the subsequent work.

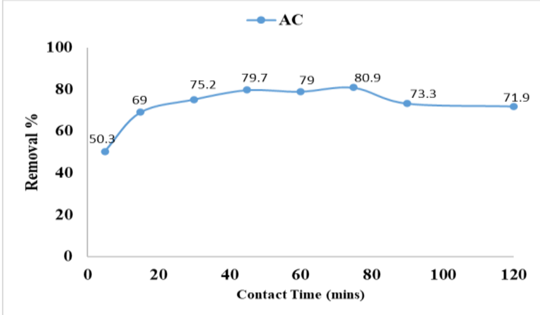

Figure 4: Effect of contact time on MB removal by AC (adsorbent dose =0.40 g, pH = 7.5 at 25◦C)

3.2.1.4 Effect of initial dye concentration

The effect of initial MB dye concentration on its removal percentage is shown Figure 5. It is observed that the removal percentage of MB dye is slightly increased at 10 ppm then decreased with increasing its concentration (20–150 ppm) in the solution. Since the available adsorption active sites remain constant but the number of dye molecules increase with increasing its concentration, this leads to the exhaustion of active sites rapidly which leads to decrease in removal percentage of the MB dye (**Singh et al., 2019**).

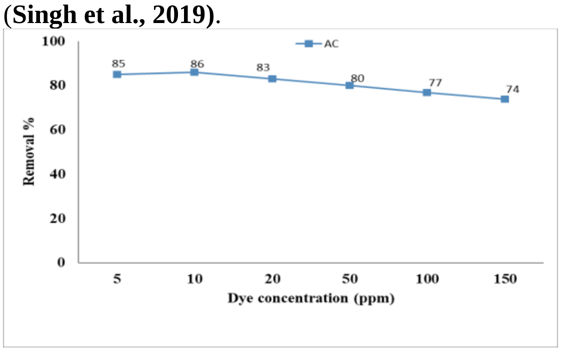

Figure 5: Effect of initial MB dye concentration on its removal by AC (adsorbent dose = 0.40 g, pH = 7.5, and contact time = 60 min at 25◦C)

4.3. Adsorption kinetics

The adsorption kinetic expresses the removal rate of adsorbate by adsorbent and the interaction between them. In the present study, the adsorption mechanism and the adsorption process parameters are estimated employing pseudo-first-order (Figure 6) and pseudo-second-order (Figure 7) kinetic models **(Labied et al., 2018).** The best suitable kinetic model that fit the experimental data is predicted from the values of correlation coefficient for the adsorption process. The kinetic models' parameters and the correlation coefficient (R^2) values of the adsorption for MB dye on AC are listed in Table 1. It is clear that the R^2 value obtained from the pseudo second-order is consistently higher (0.9991) than that achieved from the pseudo-first-order (0.8951).

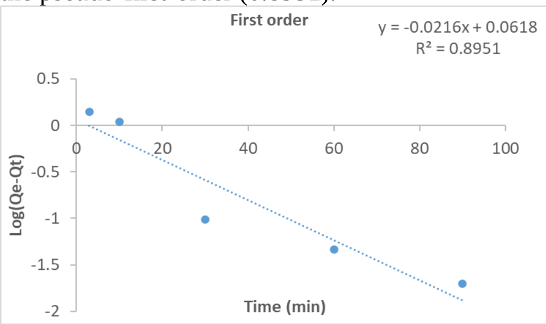

Figure 6: Pseudo-first-order for the adsorption of MB dye on AC

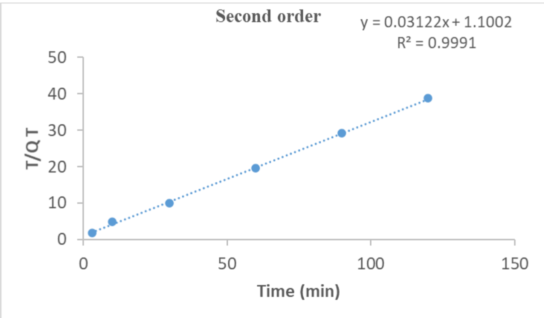

Figure 7: Pseudo-second–order for the adsorption of MB dye on AC

In addition, the q_e value coincided with the experimental q_e value (qe, exp). From the following equation, $t/q_t = t/q_e + 1/k\, q_e^2$, q_e can be calculated: $1/q_e = 0.03122$ then q_e=32.03 (calculated).

Table 1: Pseudo-first order and pseudo-second order kinetic models' parameters for the adsorption of MB by AC

Pseudo-first order constants	Pseudo-second order constants
K_1 (min^{-1}): 0.049 q_e (mg/g): 0.90 R^2: 0.8951	K_2 (g/mg/min):0.104 q_e (mg/g): 32.03 R^2:0.9991

This means that the pseudo-second order kinetic model for the adsorption of MB dye on AC is fitting more than that of pseudo first order. This suggests that the controlling rate step between adsorbent and adsorbate is chemisorption and the rate of adsorption depended on the availability of active sites on the surface of adsorbent rather than MB concentration in bulk solution **(Liu, 2008).** Similar results have been reported **(Singh et al., 2019)** for uptake of MB onto activated carbon prepared from biowaste precursor. Also, the obtained results are in a good agreement with the results of **Abuzerr et al., 2018.** who stated that the adsorption of MB at all concentrations fitted with the pseudo-second-order **(Abuzerr et al., 2018)**. In another study by **Geçgel et al ., 2012,** it has been reported that the kinetic of adsorption process was found to follow the pseudo second- order kinetic model for removal of methylene blue using carbon prepared from Pea Shells (**Gao et al., 2013).**

3.4 Adsorption Isotherms

The adsorption isotherm describes how the adsorbed molecules are distributed between the liquid and the adsorbent at equilibrium. It correlates the relationship between equilibrium concentration (C_e) of adsorbate and its amount of uptake per unit mass of the adsorbent (Q_e). In order to identify the best model fitting the experimental data, the most two common isotherms models; namely, Langmuir and Freundlich were examined **(Kuang et al., 2020).**

Table 2: Langmuir and Freundlich parameters for adsorption of MB dye on AC

Langmuir isotherm		Freundlich isotherm	
Q (mg/g)	7.2	**Kf**	0.87
b (L/mg)	2.5	**n**	0.46
R^2	0.9887		
R_L	0.008	**R^2**	0.944

The Langmuir model postulates that the adsorption occurs at specific homogeneous sites on the adsorbent and is used successfully in many monolayer adsorption processes**.** The Freundlich model endorses the heterogeneity of the surface and assumes that the adsorption occurs at sites with different energy levels of adsorption **(Geçgel et al., 2012).** The applicability of the isotherm equations is compared by judging the correlation coefficients (R^2).

The linear form of Langmuir isotherm equation is:

$$\frac{Ce}{qe} = \frac{1}{Qob} + \frac{Ce}{Qo}$$

Where Ce is the equilibrium concentration of the dye solution (mg/ L) and qe is the amount of dye adsorbed per unit mass of adsorbent (mg/g).

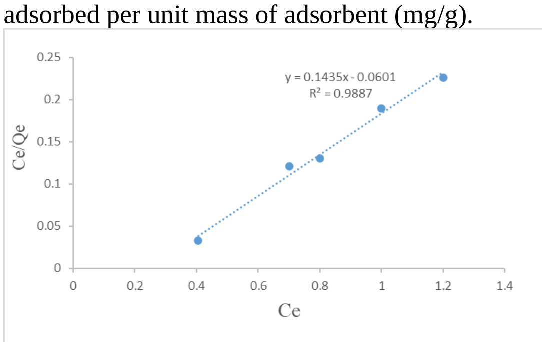

Figure 8: Langmuir isotherm plots for adsorption of MB on AC

The values Qo (mg/g) and b (L/mg) are the Langmuir constants related to the maximum adsorption capacity and the rate of adsorption, respectively.

Figure 8 shows the plots of Ce/qe versus Ce for the adsorption of MB on the AC giving a straight line of slope ($1/\,Qo$) and intercept ($1/\,Qob$). The essential characteristics of Langmuir isotherm can be expressed in terms of dimensionless equilibrium parameter (RL). The parameter is defined by:

$$Rl = \frac{1}{1 + bCo}$$

Where b is the Langmuir constant and Co is the maximum dye concentration (mg L^{-1}). The value of RL indicates the type of the isotherm to be unfavourable (RL > 1), linear (RL =1), favourable (0 < RL < 1), and irreversible (RL = 0).

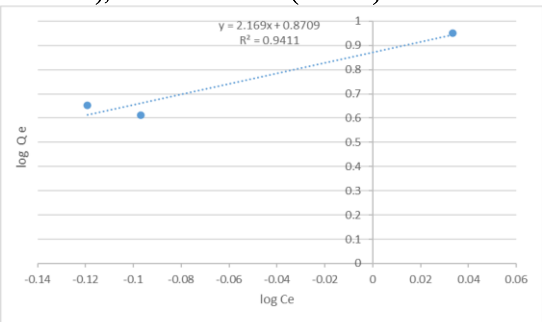

Figure 9: Freundlich isotherm plots for adsorption of MB on AC

The logarithmic form of Freundlich model is given by the following equation:

$$\ln qe = \ln Kf + \frac{1}{n} \ln Ce$$

Where, KF (mg g^{-1}) *(L $mg^{-1})^{1/n}$ and $1/n$ are Freundlich constants related to adsorption capacity and adsorption intensity of the adsorbent, respectively. The values KF and $1/n$ are obtained from the intercepts (ln K_F) and slope ($1/n$) of the linear plots of ln q_e versus ln C_e (Figure 9). The slope $1/n$ is a measure of adsorption intensity or surface heterogeneity as the adsorbent surface becomes more heterogeneous if the value of $1/n$ closes to zero, meanwhile, if the value of $1/n$ below one this indicates a normal Freundlich isotherm and if it is above one this is indicative of cooperative adsorption **(Zhang et al., 2010).**

Table 2 displays the parameters values of the two isotherms and related correlation coefficients. The comparison of correlation coefficients (R^2) of the linearized form of both equations indicates that the Langmuir model yields a better fit for the experimental equilibrium adsorption data than the Freundlich model with the maximum adsorption capacity (Q_o) value determined as 7.2 mg/ g^{-1} at 25 ◦C, It is clear that the values of R_L was in the range of 0-1 and this conformed that the Langmuir isotherm was favourable for adsorption of MB on AC.

Conclusion

The followings are the concluded remarks

- The activated carbon has been successfully prepared by thermal and chemical activations of rice husk. The prepared AC was successfully used to remove the MB from wastewater by adsorption process. The maximum adsorption process reached more than 80%.

In the adsorption process, the reaction proceeded by pseudo–second order model and the Langmuir isotherm model was the predominant mechanism with a maximum adsorption.

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